

Supporting Information

Synthesis of 3,4,5-trisubstituted isoxazoles via 1,3-dipolar cycloaddition/SO₂ extrusion of benzoisothiazole-2,2-dioxide-3-ylidenes with nitrile oxides

*Guorui Cao,^a Yu Wang,^a Tao Cui,^a Longjiang Huang,^{ab} Dawei Teng^{*a}*

^aCollege of Chemical Engineering, Qingdao University of Science and Technology, 53 Zhengzhou Lu, Qingdao 266042, China

^bState Key Laboratory of Bioactive Substance and Function of Natural Medicines, Institute of Materia Medica, Chinese Academy of Medical Sciences and Peking Union Medical College, Beijing 100050, P. R. China

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General

All the reagents were purchased from TCI chemicals and local suppliers and used without purification. The starting materials **1** were prepared following our previous paper¹. All reactions were monitored by TLC. Chromatography refers to open column chromatography on silica gel (100-200 mesh).

¹H NMR spectra were recorded on 500 MHz and ¹³C NMR spectra were recorded on 125 MHz by using a Bruker Avance 500M spectrometer. Chemical shifts were reported in parts per million (δ) relative to tetramethylsilane (TMS). Mass spectra were performed on an Ultima Global spectrometer with an ESI source. The X-ray single-crystal diffraction was performed on Saturn 724+ instrument.

General procedure for the preparation of 3,4,5-trisubstituted isoxazoles **4**

To a mixture of (Z)-benzothiazole-2,2-dioxide-3-ylidenes **1** (0.2 mmol) and powdered 4Å MS (0.11g, 0.6 mmol) in toluene (1 mL), nitrile oxide precursors **2** (0.6 mmol) was added slowly during 24 hours. After that, the mixture was filtered and the filtrate was evaporated to remove solvent under reduced pressure.. The residue was subjected to column chromatography on silica gel (100-200 mesh) using petroleum/dichloromethane as eluent to afford dipolarophiles **4**.

{2-[3-(4-Methoxy-phenyl)-4-phenyl-isoxazol-5-yl]-phenyl}-methyl-amine (4o) (CCDC 1441667)

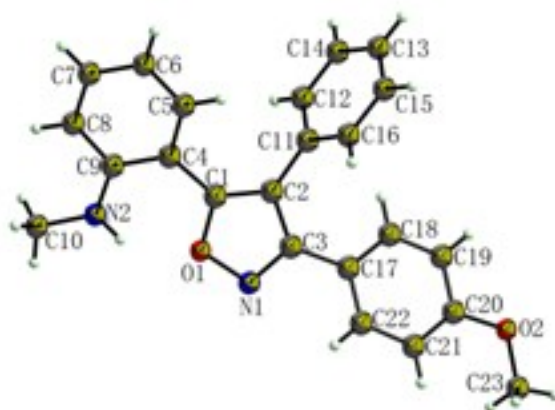
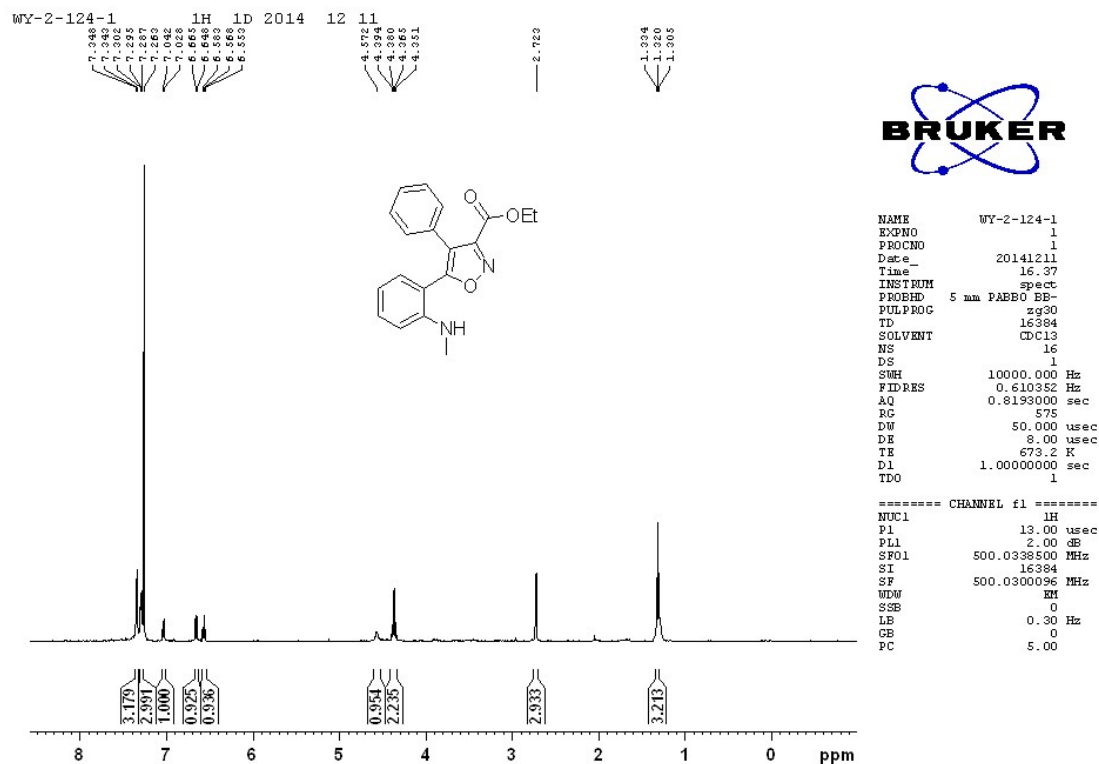


Fig 2. X-ray crystallography of compound **4o**.

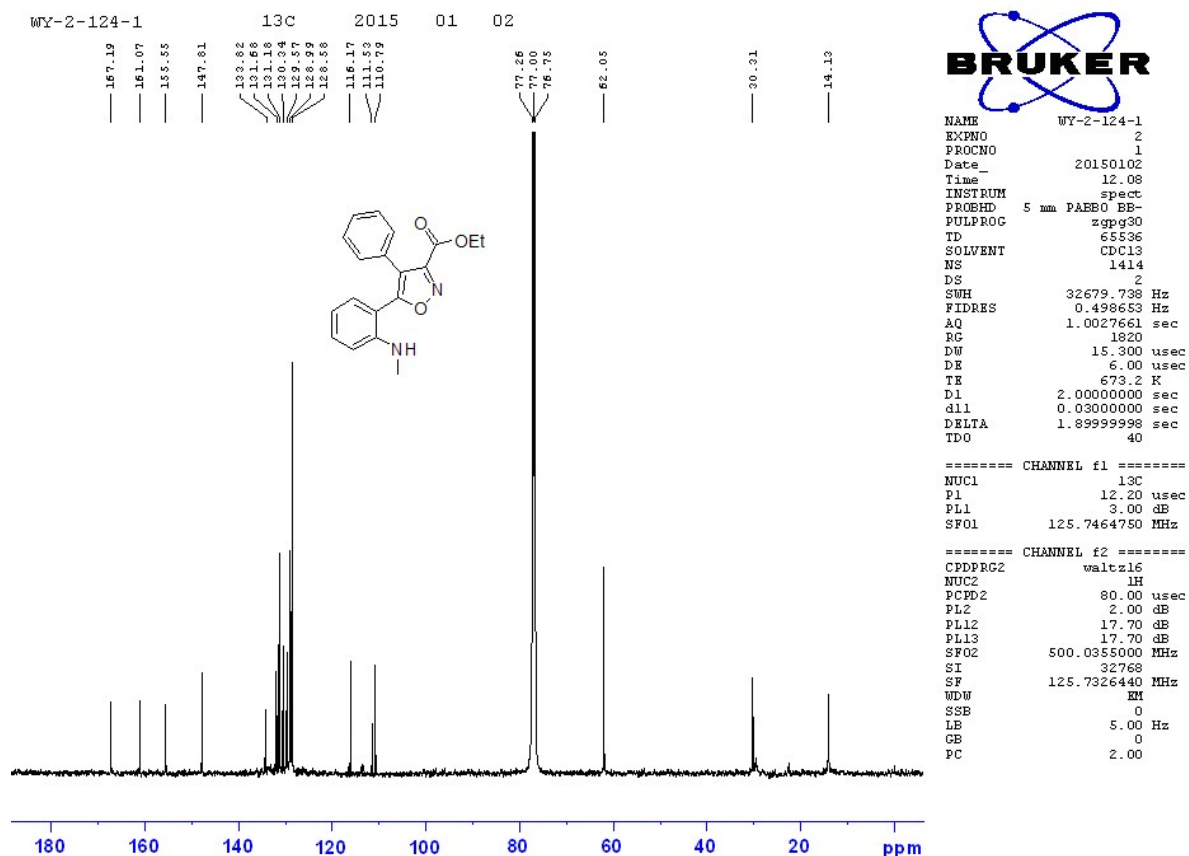
1. Cao, G.; Long, F.; Zhao, Y.; Wang, Y.; Huang, L.; Teng D. *Tetrahedron*, **2014**, 70, 9359-9365.

Characterization spectra of compounds 4

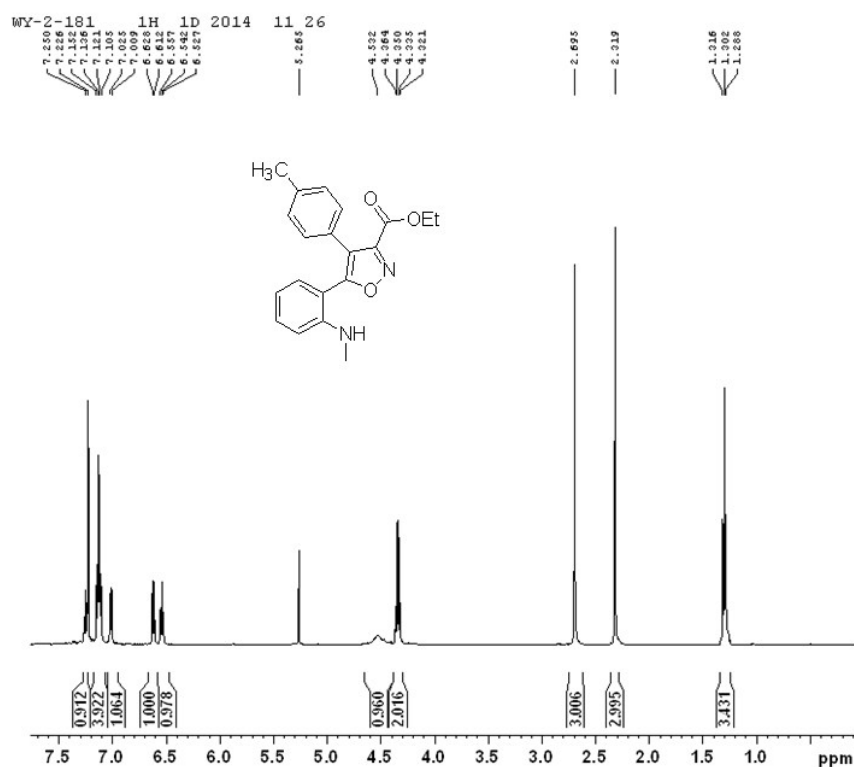
¹H NMR Spectrum of 4a



¹³C NMR Spectrum of 4a



¹H NMR Spectrum of 4b

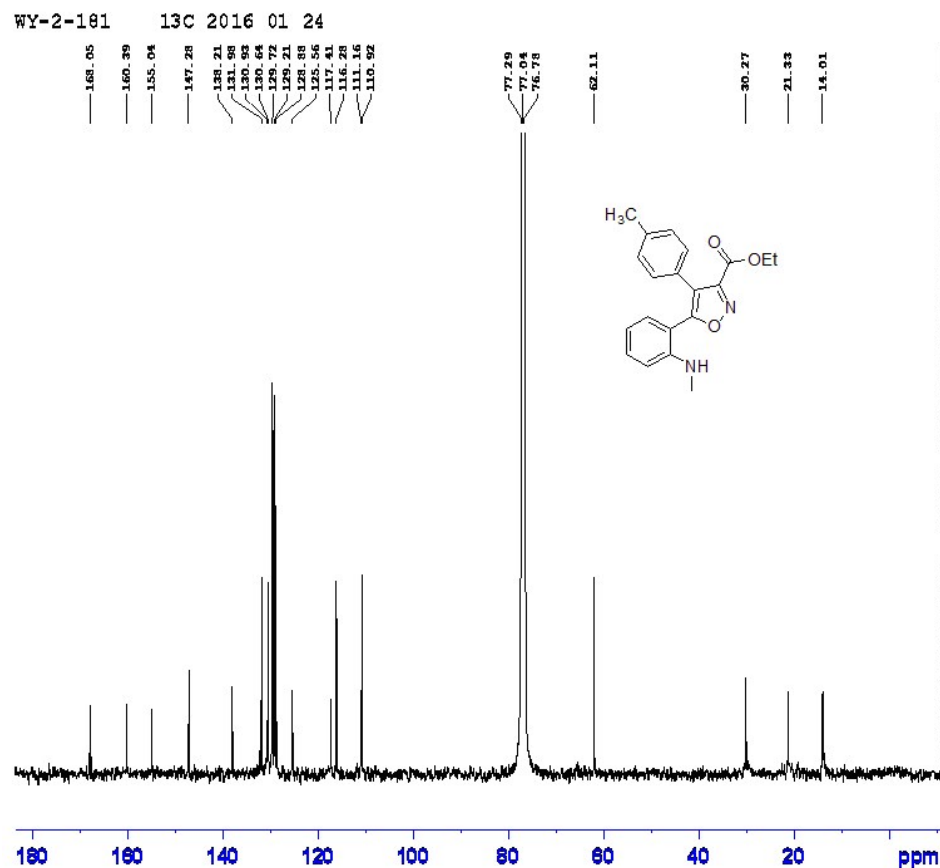


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TD         16384
SOLVENT    CDCl3
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DS          1
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FIDRES      0.610352 Hz
AQ          0.8193000 sec
RG          575
DW          50.000 usec
DE          8.00 usec
TE          673.2 K
D1          1.00000000 sec
TD0         1

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¹³C NMR Spectrum of 4b



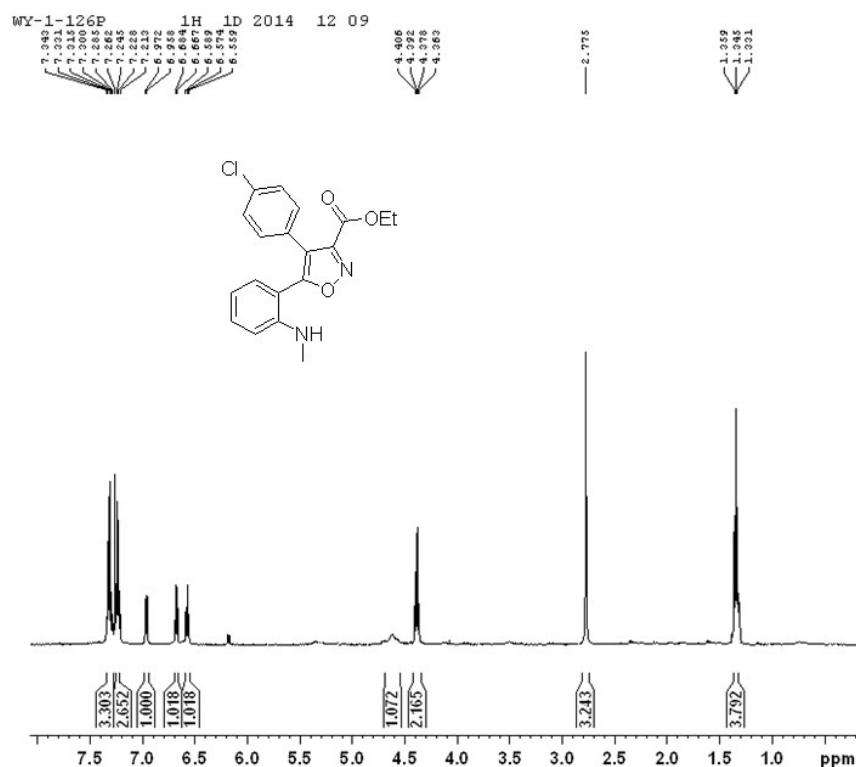
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FIDRES      0.498653 Hz
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RG          1060
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TE          673.2 K
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P1         12.20 usec
PL1        3.00 dB
SF01       125.7464750 MHz

===== CHANNEL f2 =====
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NUC2       1H
PCPD2      80.00 usec
PL2        2.00 dB
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PL13       17.70 dB
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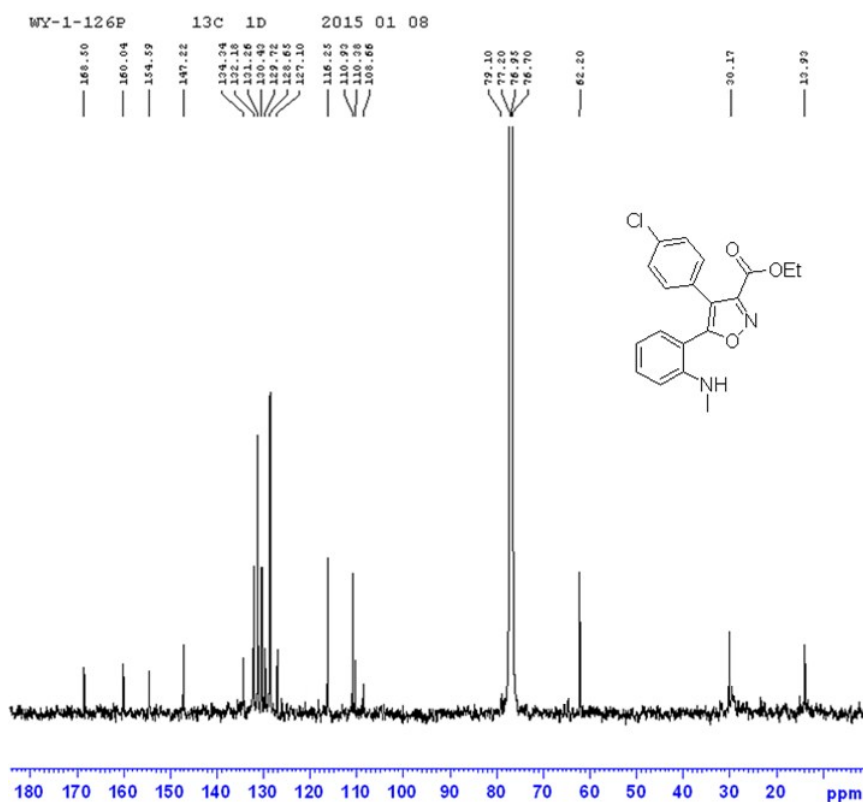
¹H NMR Spectrum of 4c



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PULPROG zg30
TD 16384
SOLVENT CDCl3
NS 16
DS 1
SWH 10000.000 Hz
FIDRES 0.610352 Hz
AQ 0.8193000 sec
RG 28.5
DW 50.000 usec
DE 8.00 usec
TE 673.2 K
D1 1.0000000 sec
TD0 1

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PL1 2.00 dB
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SSB 0
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GB 0
PC 2.00

¹³C NMR Spectrum of 4c



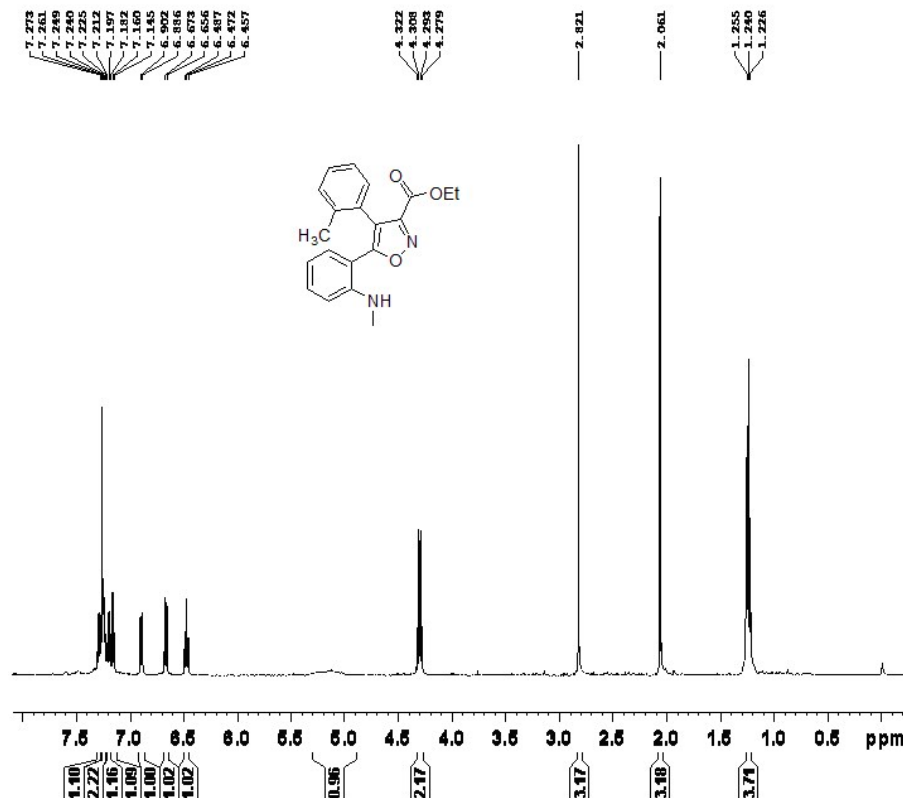
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NS 3485
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FIDRES 0.498653 Hz
AQ 1.0027661 sec
RG 1290
DW 15.300 usec
DE 6.00 usec
TE 291.6 K
D1 2.00000000 sec
d11 0.03000000 sec
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TD0 20

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SF01 125.7464750 MHz

===== CHANNEL f2 =====
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NUC2 1H
PCPD2 80.00 usec
PL2 2.00 dB
PL12 17.70 dB
PL13 17.70 dB
SF02 500.0355000 MHz
SI 32768
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SSB 0
LB 8.00 Hz
GB 0
PC 2.00

¹H NMR Spectrum of 4d

WY-2-130 1H 1D 2016 01 23

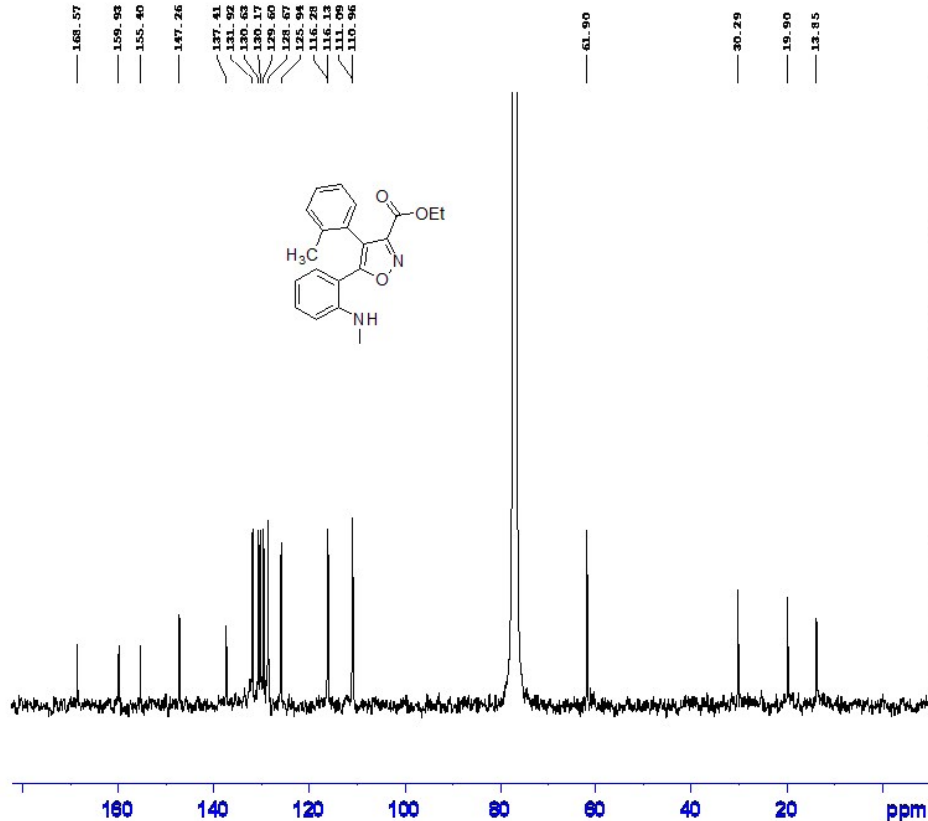


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TD         16384
SOLVENT   CDCl3
NS         16
DS         1
SWH        10000.000 Hz
FIDRES     0.610352 Hz
AQ         0.6193000 sec
RG         536
DW         50.000 usec
DE         8.00 usec
TE         673.2 K
D1         1.0000000 sec
TD0        1
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NUC1       1H
P1         13.00 usec
PL1        2.00 dB
SFO1       500.0336500 MHz
SI         16384
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WDW        EM
SSB        0
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GB         0
PC         2.00
    
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¹³C NMR Spectrum of 4d

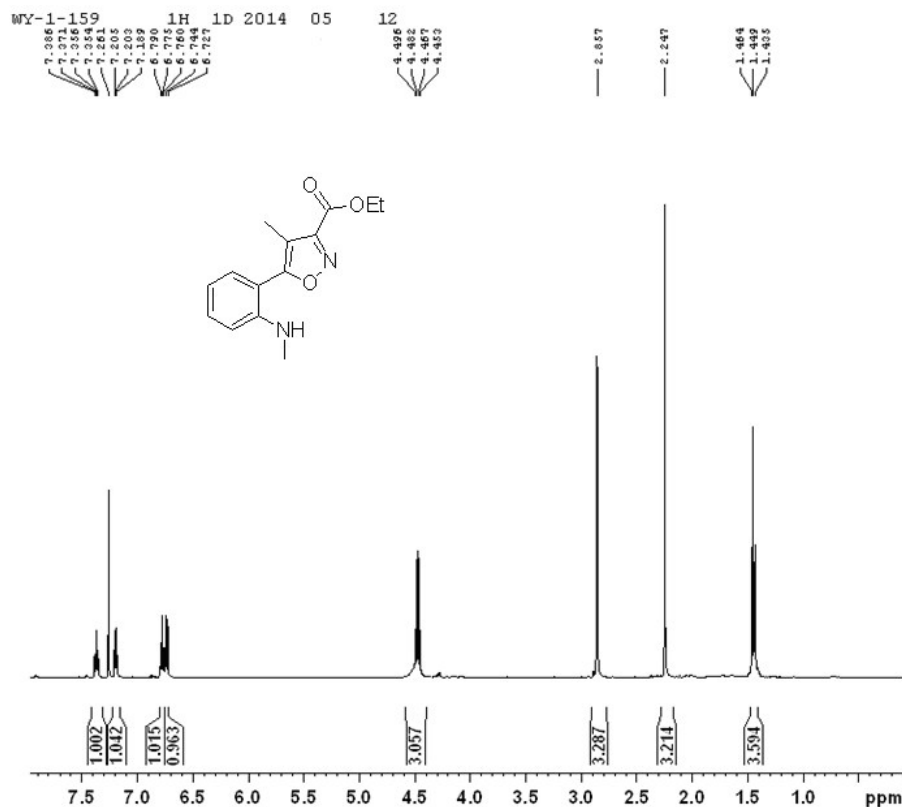
WY-2-130 13C 2016 01 26



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PROCNO    1
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PULPROG   zgpg30
TD         65536
SOLVENT   CDCl3
NS         14322
DS         2
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FIDRES     0.496653 Hz
AQ         1.0027661 sec
RG         2231
DW         15.300 usec
DE         6.00 usec
TE         673.2 K
D1         2.0000000 sec
d11        0.0300000 sec
DELTA      1.8999999 sec
TD0        40
----- CHANNEL f1 -----
NUC1       13C
P1         12.20 usec
PL1        3.00 dB
SFO1       125.7464750 MHz
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CFDPG2     waltz16
NUC2       1H
PCPD2      80.00 usec
PL2        2.00 dB
PL12       17.70 dB
PL13       17.70 dB
SFO2       500.0355000 MHz
SI         32768
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PC         2.00
    
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¹H NMR Spectrum of 4e

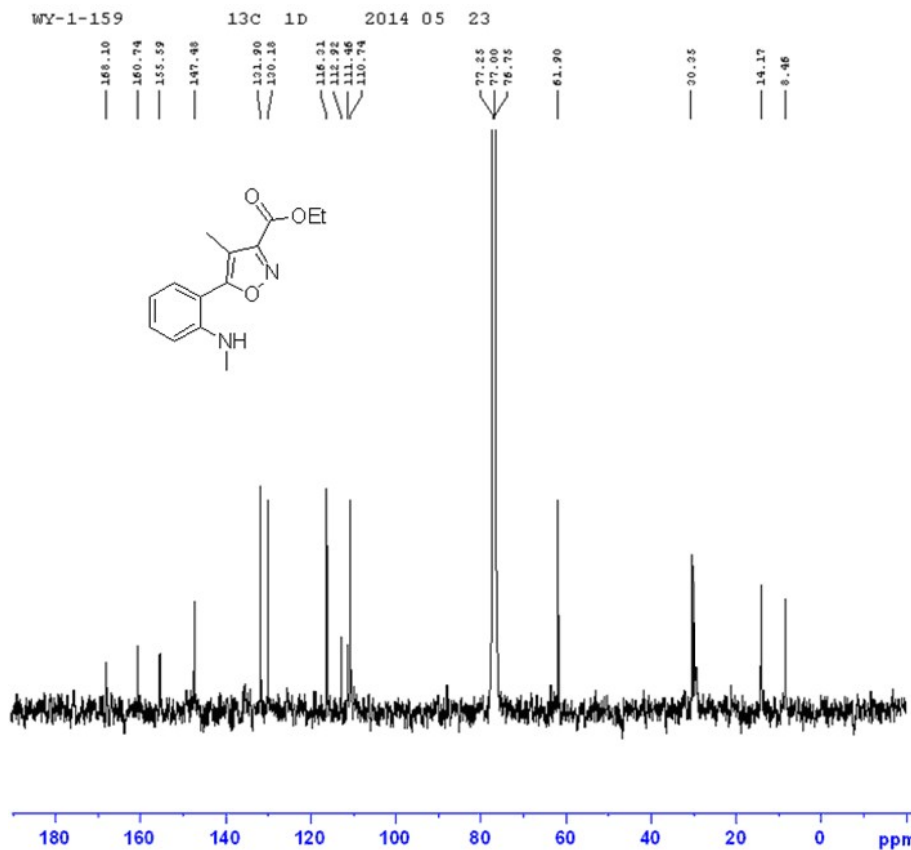


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PULPROG   zg30
TD         16384
SOLVENT   CDCl3
NS         8
DS         1
SWH        10000.000 Hz
FIDRES     0.610352 Hz
AQ         0.8193000 sec
RG         1150
DW         50.000 usec
DE         8.00 usec
TE         297.5 K
D1         1.00000000 sec
TD0        1

===== CHANNEL f1 =====
NUC1       1H
P1         13.00 usec
PL1        2.00 dB
SFO1       500.0338500 MHz
SI         16384
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SSB        0
LB         0.30 Hz
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PC         4.00
  
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¹³C NMR Spectrum of 4e



```

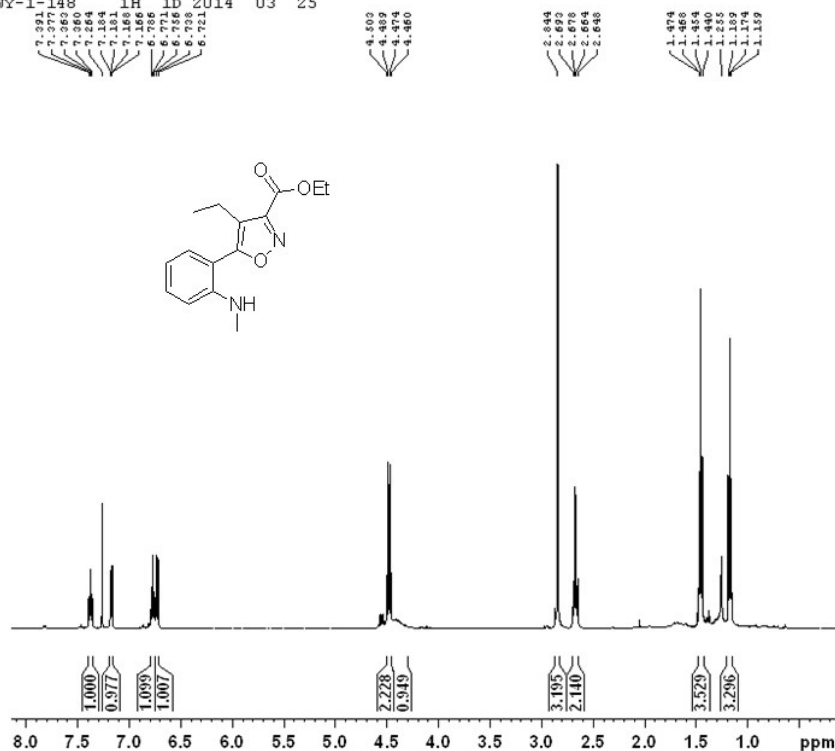
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SOLVENT   CDCl3
NS         1640
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AQ         1.0027661 sec
RG         1820
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DE         6.00 usec
TE         299.3 K
D1         2.00000000 sec
d11        0.03000000 sec
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TD0        20

===== CHANNEL f1 =====
NUC1       13C
P1         12.20 usec
PL1        3.00 dB
SFO1       125.7464750 MHz

===== CHANNEL f2 =====
CPDPRG2   waltz16
NUC2       1H
PCPD2      80.00 usec
PL2        2.00 dB
PL12       17.70 dB
PL13       17.70 dB
SFO2       500.0355000 MHz
SI         32768
SF         125.7326454 MHz
WDW        EM
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¹H NMR Spectrum of 4f

WY-1-148 1H 1D 2014 03 25

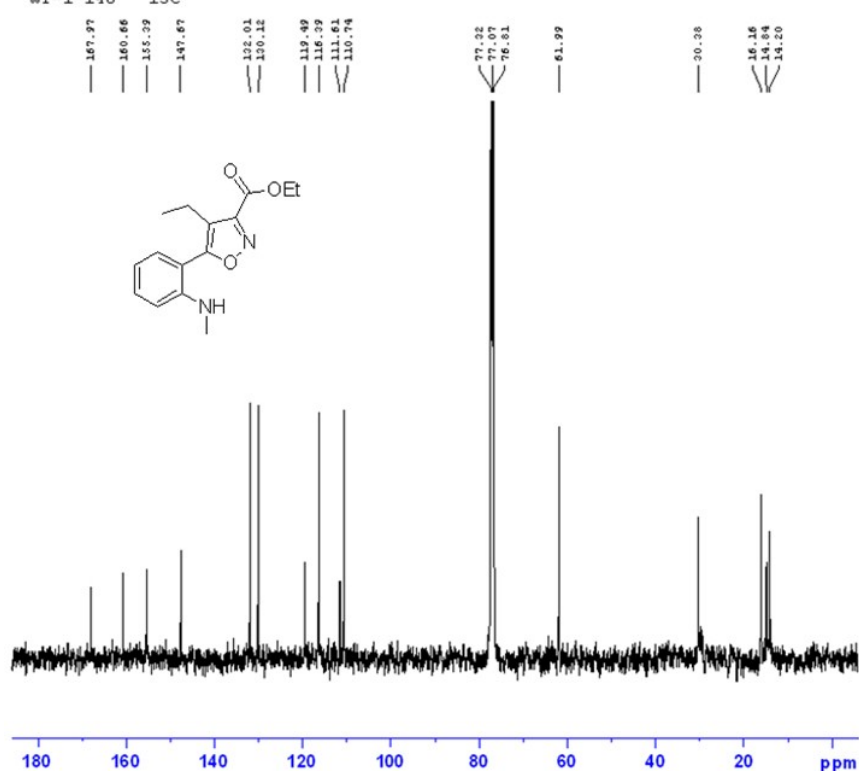


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PROCNO 1
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PULPROG zg30
TD 16384
SOLVENT CDCl3
NS 36
DS 1
SWH 10000.000 Hz
FIDRES 0.610352 Hz
AQ 0.8193000 sec
RG 287
DW 50.000 usec
DE 8.00 usec
TE 673.2 K
DL 1.0000000 sec
TDO 1

===== CHANNEL f1 =====
NUC1 1H
P1 13.00 usec
PL1 2.00 dB
SF01 500.038500 MHz
SI 16384
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WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 4.00

¹³C NMR Spectrum of 4f

WY-1-148 13C



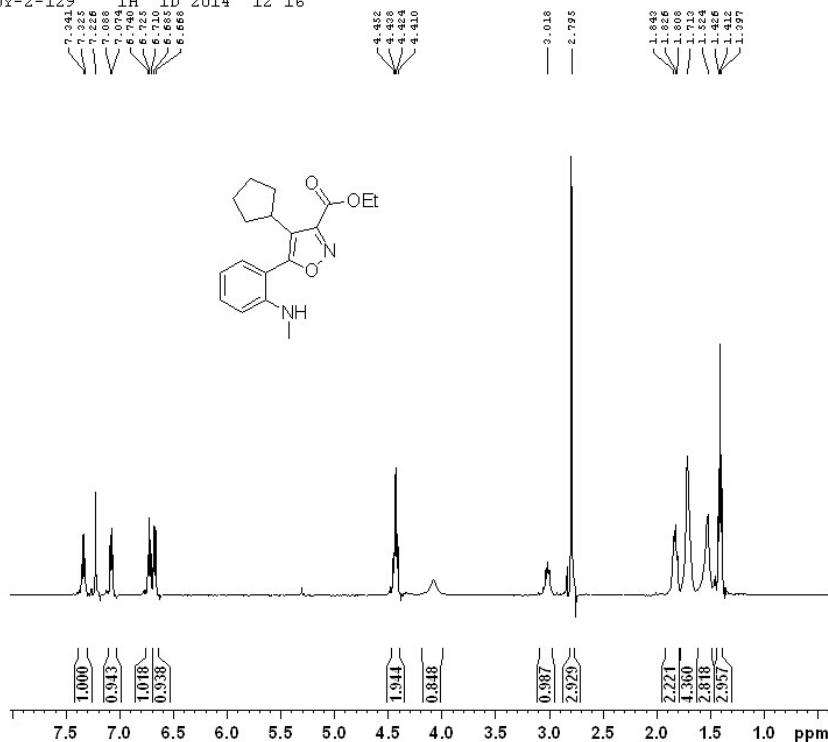
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SOLVENT CDCl3
NS 716
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FIDRES 0.498653 Hz
AQ 1.0027661 sec
RG 2300
DW 15.300 usec
DE 6.00 usec
TE 673.2 K
DL 2.0000000 sec
dL1 0.03000000 sec
DELTA 1.89999998 sec
TDO 1

===== CHANNEL f1 =====
NUC1 13C
P1 12.20 usec
PL1 3.00 dB
SF01 125.7464750 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.00 dB
PL12 17.70 dB
PL13 17.70 dB
SF02 500.0355000 MHz
SI 32768
SF 125.7326392 MHz
WDW EM
SSB 0
LB 6.00 Hz
GB 0
PC 1.00

¹H NMR Spectrum of 4g

WY-2-129 1H 1D 2014 12 16



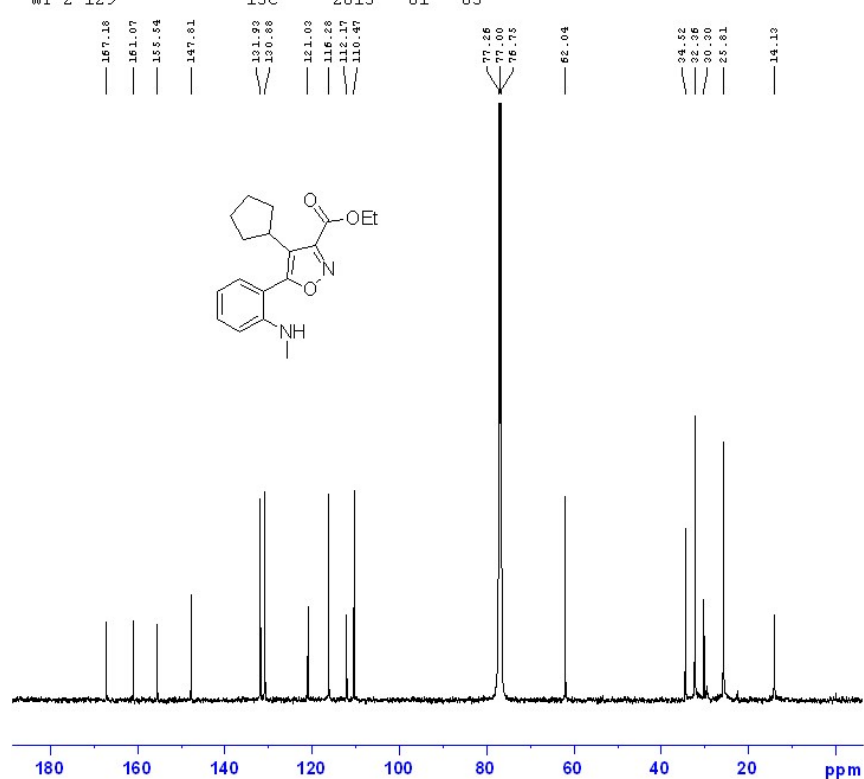
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NS         8
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SWH        10000.000 Hz
FIDRES     0.610352 Hz
AQ         0.8193000 sec
RG         18
DW         50.000 usec
DE         8.00 usec
TE         673.2 K
D1         1.00000000 sec
TD0        1

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PL1        2.00 dB
SF01       500.038500 MHz
SI         16384
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SSB        0
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¹³C NMR Spectrum of 4g

WY-2-129 13C 2015 01 05



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NAME      WY-2-129
EXPNO     2
PROCNO    1
Date_     20150105
Time      22.08
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PULPROG   zgpg30
TD         65536
SOLVENT   CDCl3
NS         14289
DS         2
SWH        32679.738 Hz
FIDRES     0.498653 Hz
AQ         1.0027661 sec
RG         3250
DW         15.300 usec
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D1         2.00000000 sec
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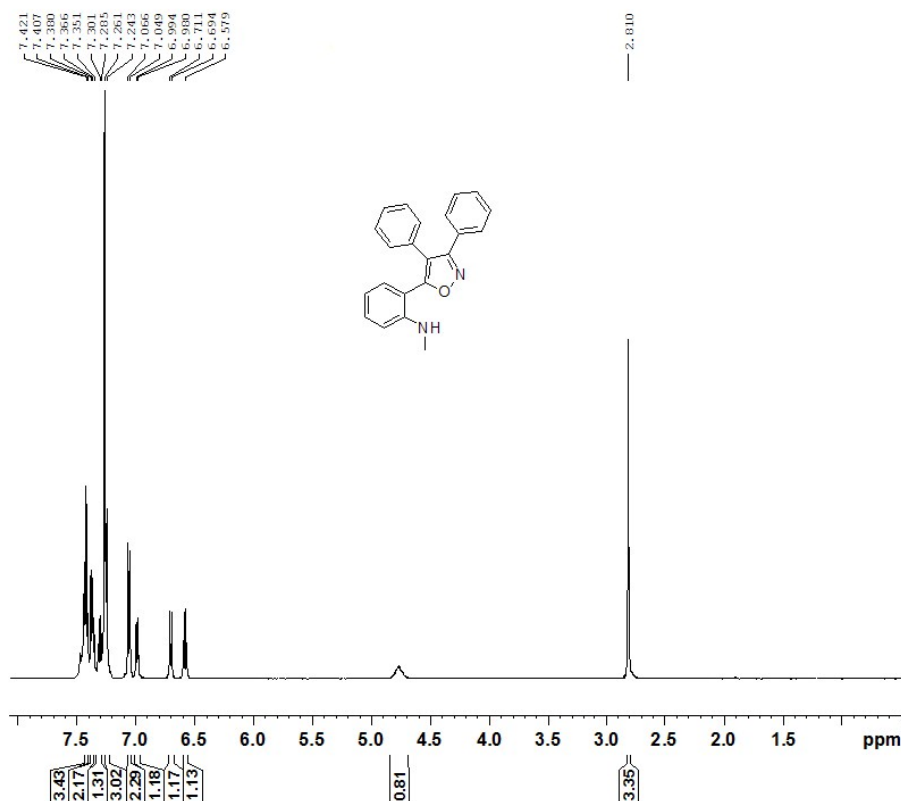
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PL2        2.00 dB
PL12       17.70 dB
PL13       17.70 dB
SF02       500.0385000 MHz
SI         32768
SF         125.7326440 MHz
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LB         5.00 Hz
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¹H NMR Spectrum of 4k

CAO-150508

1H 2016 01 25



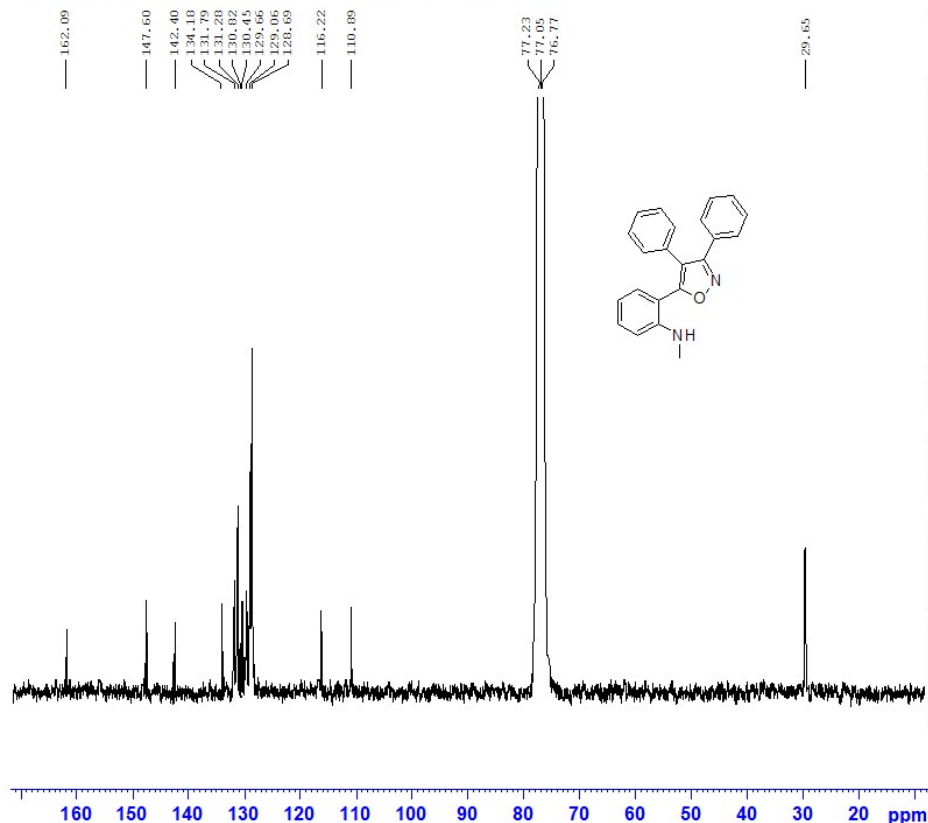
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PROCNO 1
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PULPROG zg30
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NS 16
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FIDRES 0.305176 Hz
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RG 907
DW 50.000 usec
DE 6.00 usec
TE 673.2 K
D1 1.00000000 sec
TD0 1

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P1 13.00 usec
PL1 2.00 dB
SFO1 500.0335010 MHz
SI 16384
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GB 0
PC 4.00

¹³C NMR Spectrum of 4k

CAO-150508

13C 2016 01 25

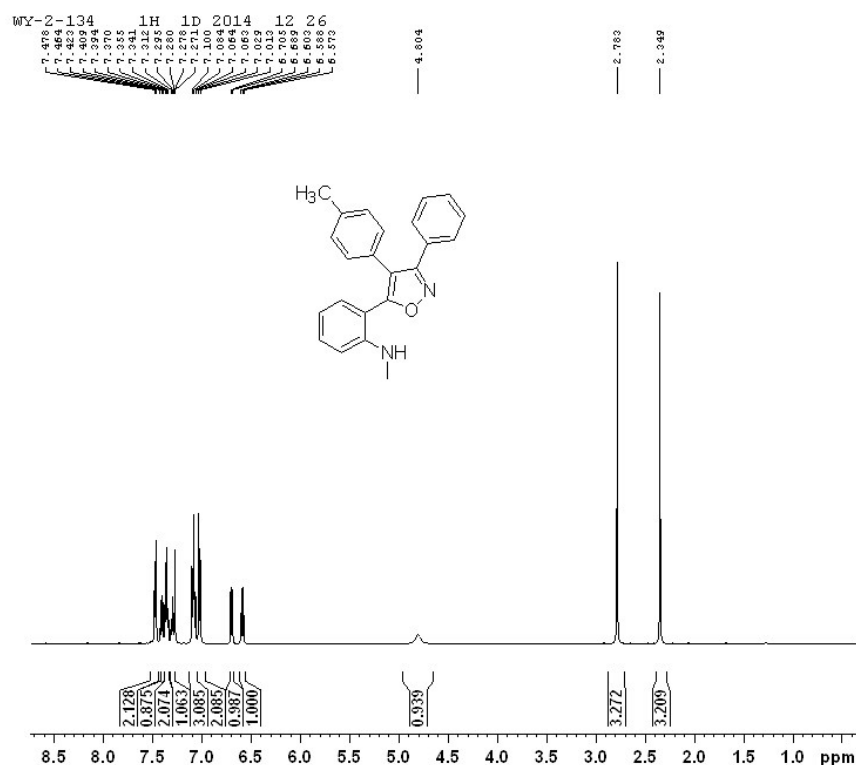


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SOLVENT CDCl3
NS 14326
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SWH 32679.738 Hz
FIDRES 0.498653 Hz
AQ 1.0027661 sec
RG 533
DW 15.300 usec
DE 6.00 usec
TE 673.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 20

===== CHANNEL f1 =====
NUC1 13C
P1 12.20 usec
PL1 3.00 dB
SFO1 125.7464750 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.00 dB
PL12 17.70 dB
PL13 17.70 dB
SFO2 500.0355000 MHz
SI 32768
SF 125.7326351 MHz
WDW EM
SSB 0
LB 10.00 Hz
GB 0
PC 1.00

¹H NMR Spectrum of 4l

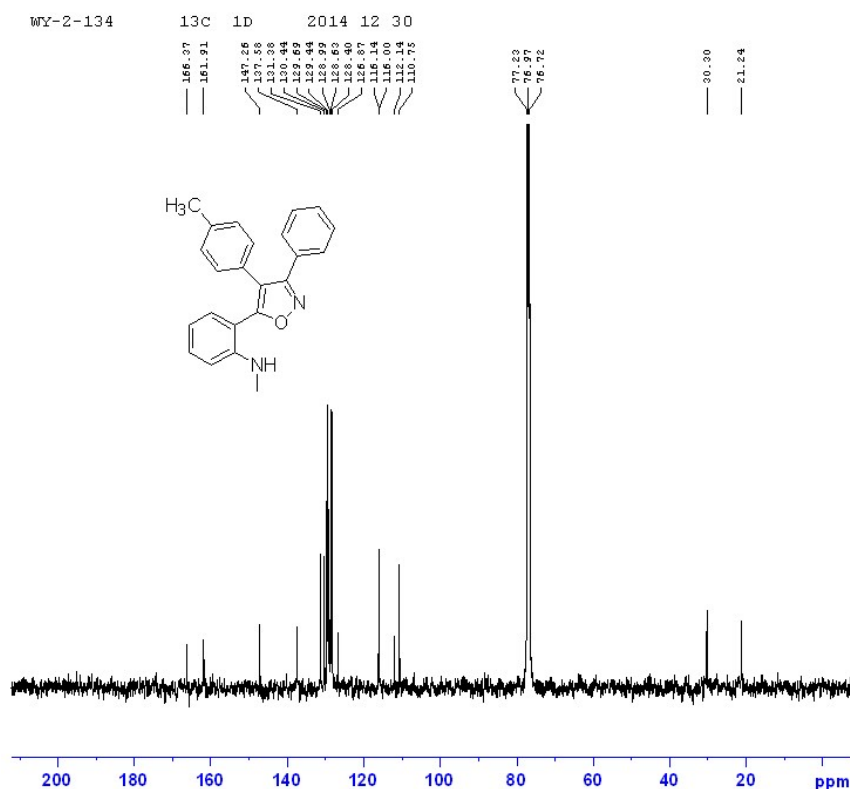


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TD         16384
SOLVENT   CDCl3
NS         8
DS         1
SWH        10000.000 Hz
FIDRES     0.610352 Hz
AQ         0.8193000 sec
RG         406
DW         50.000 usec
DE         8.00 usec
TE         673.2 K
D1         1.00000000 sec
TD0        1

===== CHANNEL f1 =====
NUC1       1H
P1         13.00 usec
PL1        2.00 dB
SF01       500.0338500 MHz
SI         16384
SF         500.0300047 MHz
WDW        EM
SSB        0
LB         0.30 Hz
GB         0
PC         2.00
  
```

¹³C NMR Spectrum of 4l



```

NAME      WY-2-134
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PROCNO    1
Date_     20141230
Time      18.31
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zgpg30
TD         65536
SOLVENT   CDCl3
NS         3283
DS         2
SWH        32679.738 Hz
FIDRES     0.498653 Hz
AQ         1.0027661 sec
RG         2890
DW         15.300 usec
DE         6.00 usec
TE         673.2 K
D1         2.00000000 sec
d11       0.03000000 sec
DELTA     1.89999998 sec
TD0        20

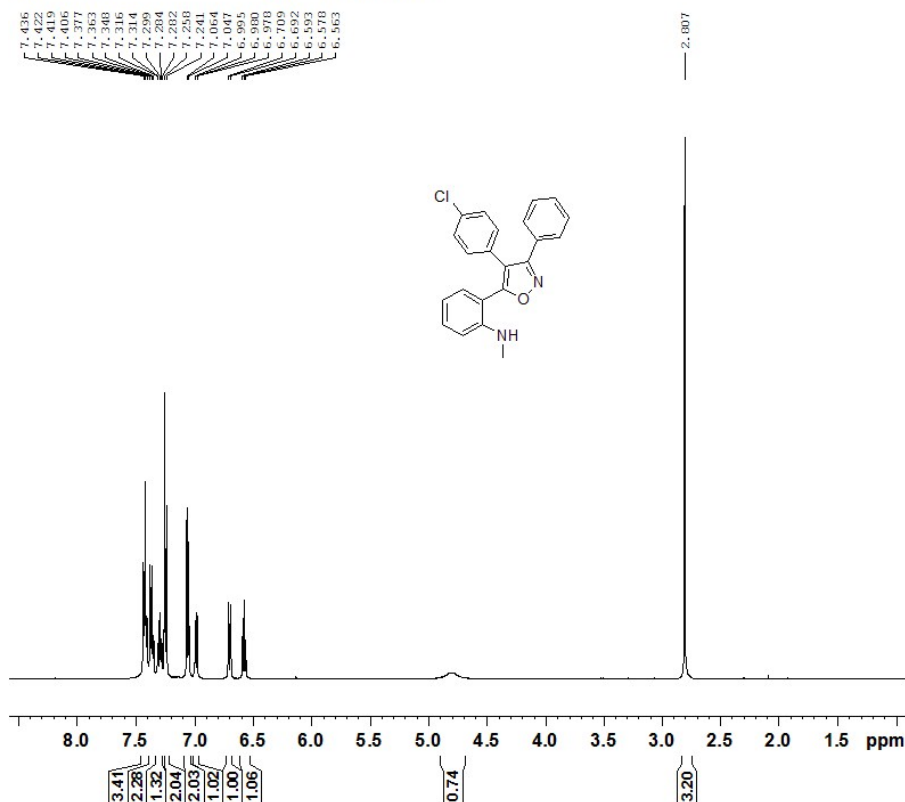
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PL1        3.00 dB
SF01       125.7464750 MHz

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NUC2       1H
PCPD2     80.00 usec
PL2        2.00 dB
PL12       17.70 dB
PL13       17.70 dB
SF02       500.0355000 MHz
SI         32768
SF         125.7326515 MHz
WDW        EM
SSB        0
LB         8.00 Hz
GB         0
PC         4.00
  
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¹H NMR Spectrum of 4m

CAO-150507

¹H 2016 01 21



```

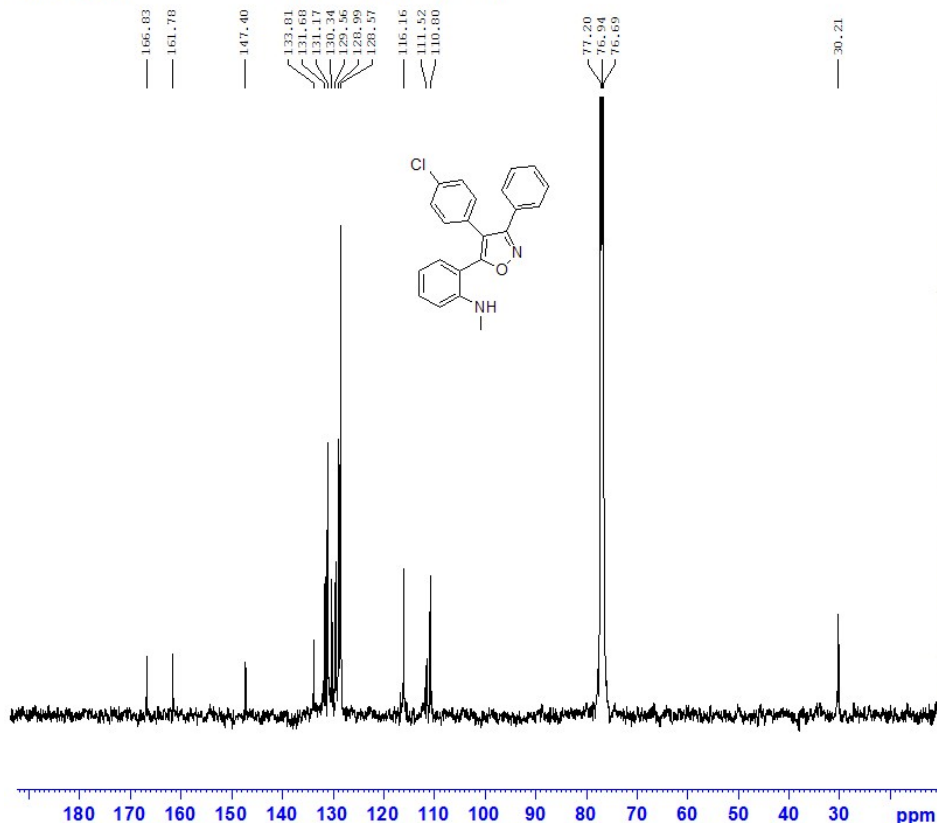
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PROCNO        1
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PULPROG       zg30
TD            32768
SOLVENT       CDCl3
NS            16
DS            1
SWH           10000.000 Hz
FIDRES        0.305176 Hz
AQ            1.6385000 sec
RG            375
DW            50.000 usec
DE            6.00 usec
TE            673.2 K
D1            1.00000000 sec
TD0           1

===== CHANNEL f1 =====
NUC1           1H
P1            13.00 usec
PL1           2.00 dB
SFO1          500.035010 MHz
SI            16384
SF            500.0300115 MHz
WDW           EM
SSB           0
LB            0.30 Hz
GB            0
PC            4.00
    
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¹³C NMR Spectrum of 4m

CAO-150507

¹³C 2016 01 21



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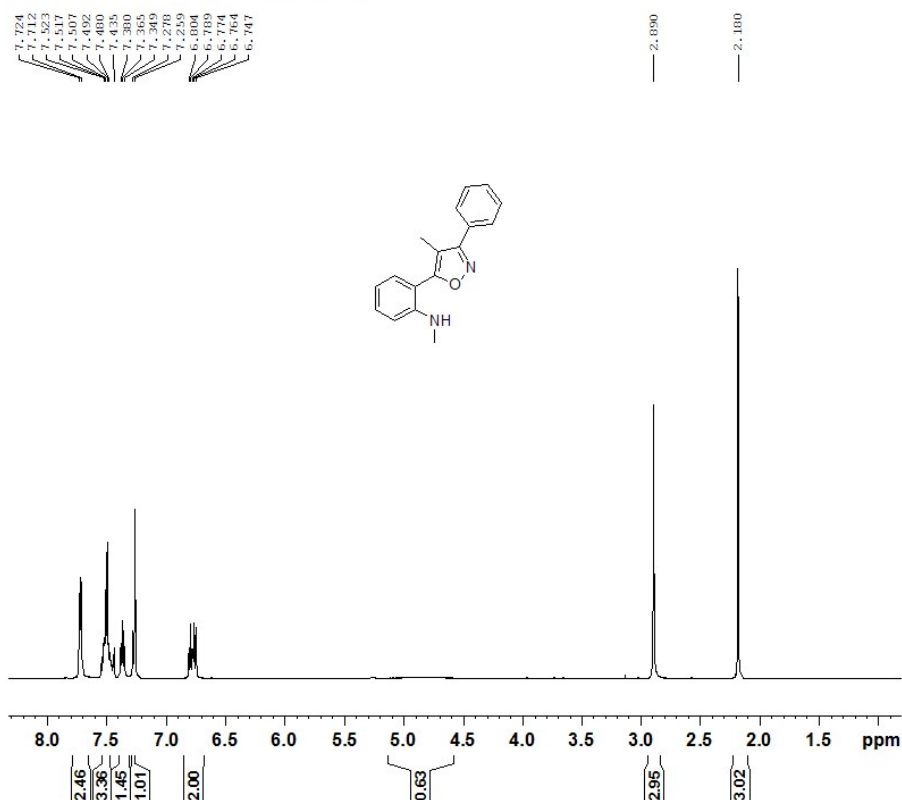
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PROCNO        1
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Time          18.10
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TD            65536
SOLVENT       CDCl3
NS            232
DS            2
SWH           32679.738 Hz
FIDRES        0.498653 Hz
AQ            1.0027661 sec
RG            435
DW            15.300 usec
DE            6.00 usec
TE            299.1 K
D1            2.00000000 sec
d11           0.03000000 sec
DELTA         1.89999998 sec
TD0           20

===== CHANNEL f1 =====
NUC1           13C
P1            12.20 usec
PL1           3.00 dB
SFO1          125.7464750 MHz

===== CHANNEL f2 =====
CPDPRG2       waltz16
NUC2           1H
PCPD2         80.00 usec
PL2           2.00 dB
PL12          17.70 dB
PL13          17.70 dB
SFO2          500.0350000 MHz
SI            32768
SF            125.7326536 MHz
WDW           EM
SSB           0
LB            10.00 Hz
GB            0
PC            1.00
    
```

¹H NMR Spectrum of 4n

wY-3-56 1H 2015 06 10



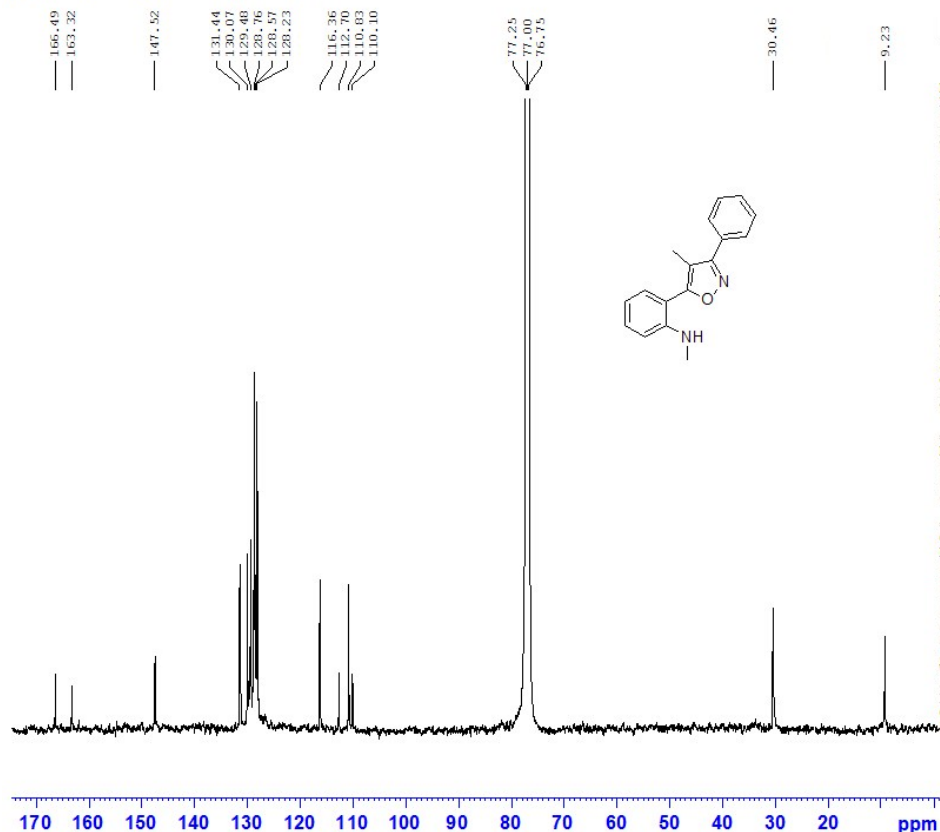
```

NAME      wY-3-56
EXPNO     1
PROCNO    1
Date_     20150610
Time      15.29
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zg30
TD         32768
SOLVENT   CDCl3
NS         16
DS         1
SWH        10000.000 Hz
FIDRES     0.305176 Hz
AQ         1.6385000 sec
RG         362
DW         50.0000 usec
DE         6.00 usec
TE         298.6 K
D1         1.00000000 sec
TD0        1

===== CHANNEL f1 =====
NUC1       1H
P1         13.00 usec
PL1        2.00 dB
SFO1       500.0335010 MHz
SI         16384
SF         500.0300113 MHz
WDW        EM
SSB        0
LB         0.30 Hz
GB         0
PC         4.00
    
```

¹³C NMR Spectrum of 4n

wY-3-56 13C 2015 06 11



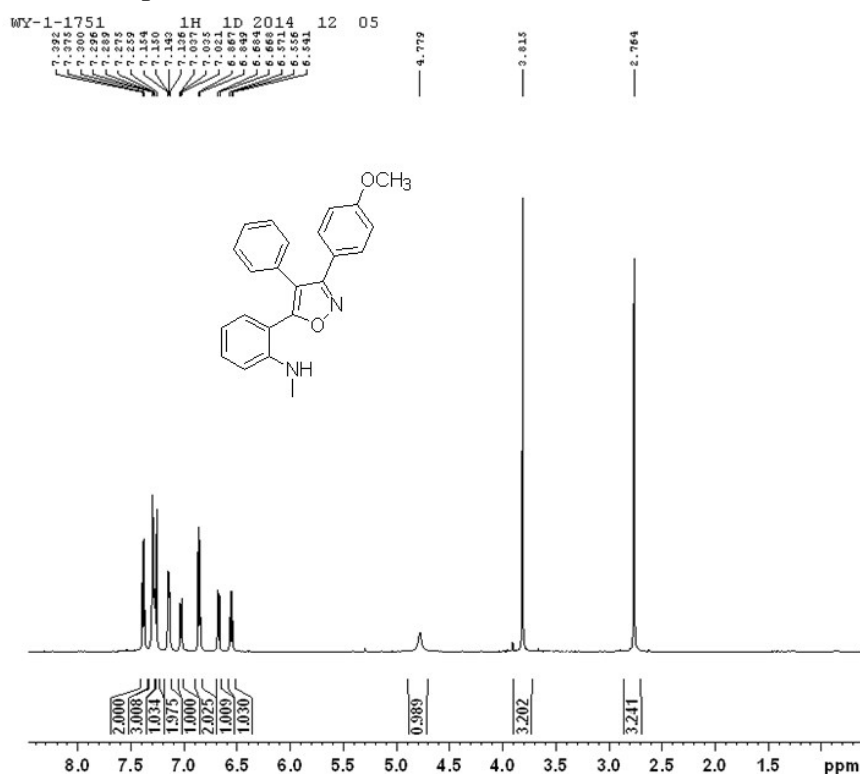
```

NAME      wY-3-56
EXPNO     2
PROCNO    1
Date_     20150611
Time      21.44
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zgpg30
TD         65536
SOLVENT   CDCl3
NS         13894
DS         2
SWH        32679.738 Hz
FIDRES     0.498653 Hz
AQ         1.0027661 sec
RG         1820
DW         15.3000 usec
DE         6.00 usec
TE         673.2 K
D1         2.00000000 sec
d11        0.03000000 sec
DELTA     1.89999998 sec
TD0        40

===== CHANNEL f1 =====
NUC1       13C
P1         12.20 usec
PL1        3.00 dB
SFO1       125.7464750 MHz

===== CHANNEL f2 =====
CPDPRG2   waltz16
NUC2       1H
PCPD2     80.00 usec
PL2        2.00 dB
PL12       17.70 dB
PL13       17.70 dB
SFO2       500.0355000 MHz
SI         32768
SF         125.7326425 MHz
WDW        EM
SSB        0
LB         10.00 Hz
GB         0
PC         0.50
    
```

¹H NMR Spectrum of 4o

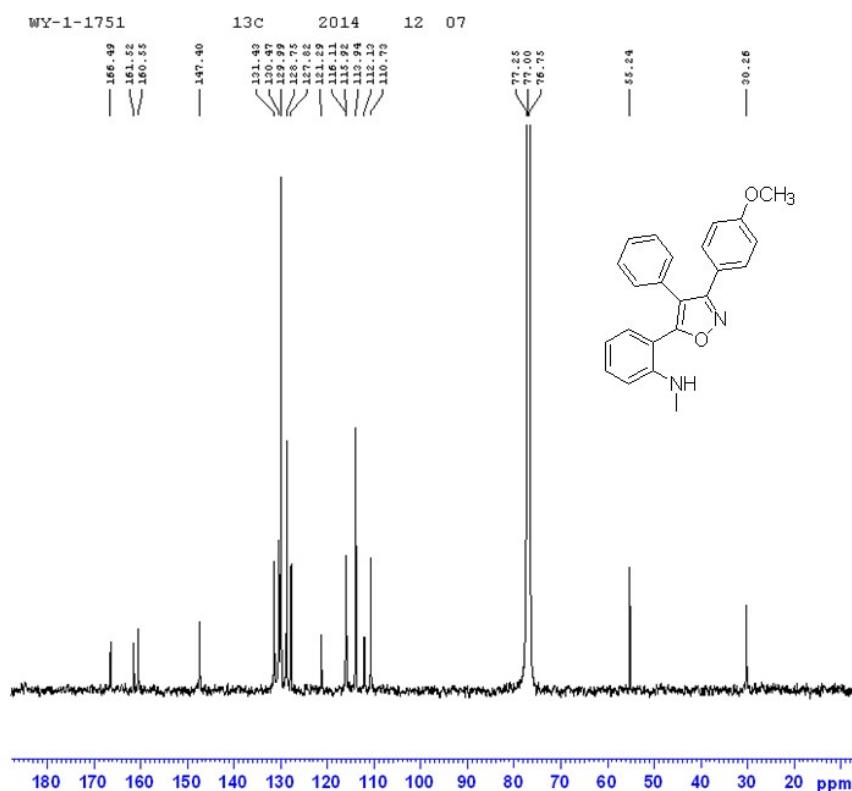


```

NAME      WY-1-1751
EXPNO     1
PROCNO    1
Date_     20141205
Time      15.48
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zg30
TD         16384
SOLVENT   CDCl3
NS         16
DS         1
SWH        10000.000 Hz
FIDRES     0.610352 Hz
AQ         0.8193000 sec
RG         406
DW         50.000 usec
DE         8.00 usec
TE         673.2 K
D1         1.00000000 sec
TD0        1

===== CHANNEL f1 =====
NUC1       1H
P1         13.00 usec
PL1        2.00 dB
SF01       500.0338500 MHz
SI         16384
SF         500.0300110 MHz
WDW        EM
SSB        0
LB         0.30 Hz
GB         0
PC         0.50
  
```

¹³C NMR Spectrum of 4o



```

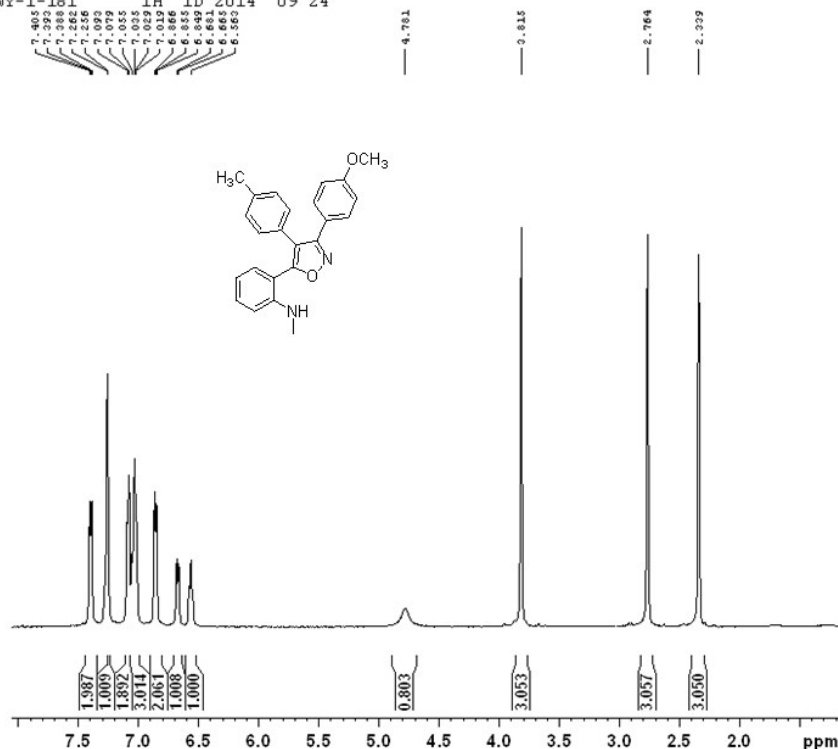
NAME      WY-1-1751
EXPNO     2
PROCNO    1
Date_     20141207
Time      12.43
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zgpg30
TD         65536
SOLVENT   CDCl3
NS         3206
DS         2
SWH        32679.738 Hz
FIDRES     0.498653 Hz
AQ         1.0027661 sec
RG         2580
DW         15.300 usec
DE         6.00 usec
TE         673.2 K
D1         2.00000000 sec
d11        0.03000000 sec
DELTA     1.89999999 sec
TD0        40

===== CHANNEL f1 =====
NUC1       13C
P1         12.20 usec
PL1        3.00 dB
SF01       125.7464750 MHz

===== CHANNEL f2 =====
CPDPRG2   waltz16
NUC2       1H
PCPD2     80.00 usec
PL2        2.00 dB
PL12       17.70 dB
PL13       17.70 dB
SF02       500.0355000 MHz
SI         32768
SF         125.7326442 MHz
WDW        EM
SSB        0
LB         10.00 Hz
GB         0
PC         2.00
  
```


¹H NMR Spectrum of 4p

WY-1-181 1H 1D 2014 09 24



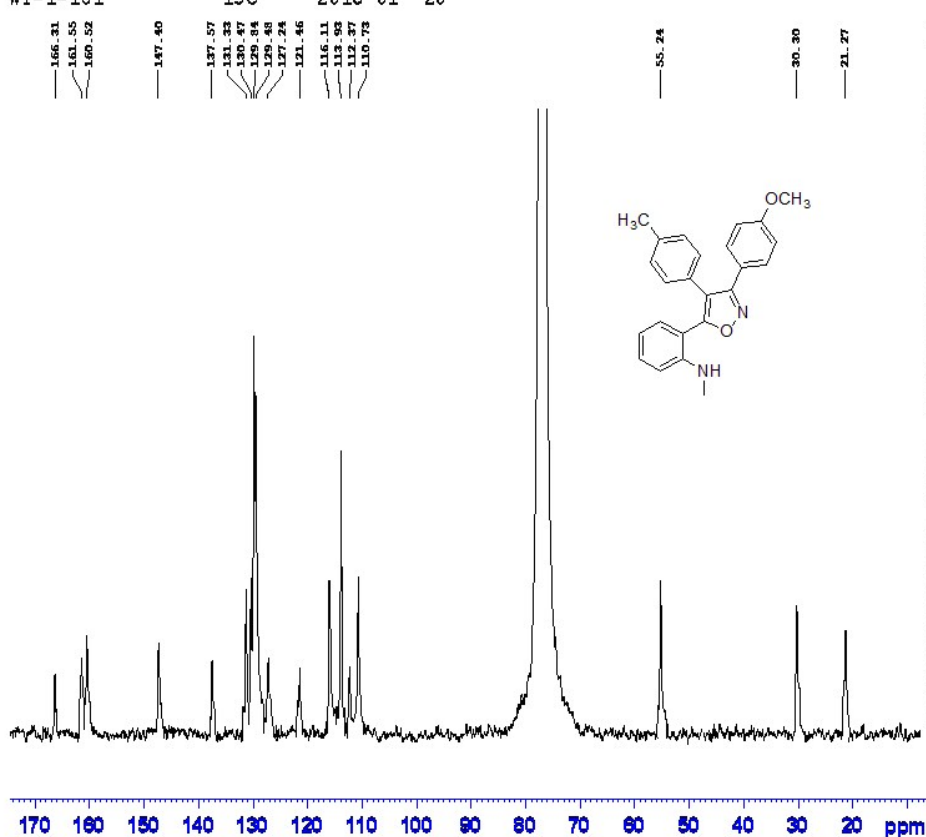
```

NAME      WY-1-181
EXPNO     11
PROCNO    1
Date_     20140916
Time      17.54
INSTRUM    spect
PROBHD     5 mm PABBO BB-
PULPROG    zg30
TD         16384
SOLVENT    CDCl3
NS         36
DS         1
SWH         10000.000 Hz
FIDRES     0.610352 Hz
AQ         0.8193000 sec
RG         912
DW         50.000 usec
DE         8.00 usec
TE         673.2 K
D1         1.00000000 sec
TDO        1

===== CHANNEL f1 =====
NUC1       1H
P1         13.00 usec
PL1        2.00 dB
SFO1       500.0338500 MHz
SI         16384
SF         500.0300120 MHz
WDW        EM
SSB        0
LB         0.50 Hz
GB         0
PC         5.00
    
```

¹³C NMR Spectrum of 4p

WY-1-181 13C 2016 01 20



```

NAME      WY-1-181
EXPNO     2
PROCNO    1
Date_     20160120
Time      15.21
INSTRUM    spect
PROBHD     5 mm PABBO BB-
PULPROG    zgpg30
TD         65536
SOLVENT    CDCl3
NS         5256
DS         2
SWH         32679.736 Hz
FIDRES     0.498653 Hz
AQ         1.0027661 sec
RG         387
DW         15.300 usec
DE         6.00 usec
TE         673.2 K
D1         2.00000000 sec
d11        0.03000000 sec
DELTA      1.65959998 sec
TDO        40

===== CHANNEL f1 =====
NUC1       13C
P1         12.20 usec
PL1        3.00 dB
SFO1       125.7464750 MHz

===== CHANNEL f2 =====
CFDPRG2    waltz16
NUC2       1H
PCPD2      80.00 usec
PL2        2.00 dB
PL12       17.70 dB
PL13       17.70 dB
SFO2       500.0355000 MHz
SI         32768
SF         125.7326420 MHz
WDW        EM
SSB        0
LB         26.00 Hz
GB         0
PC         2.00
    
```


¹H 1D 2014 11 20
 WY-1-173C1
 7.371, 7.354, 7.308, 7.305, 7.291, 7.276, 7.273, 7.268, 7.264, 7.258, 7.251, 7.077, 7.061, 7.045, 6.978, 6.962, 6.960, 6.950, 6.935, 6.922, 6.902, 6.886, 6.883, 6.868, 6.853
 4.790, 3.427, 2.405
 1.983, 3.561, 1.972, 0.984, 2.037, 1.025, 1.000, 0.924, 3.128, 3.128

CNc1ccccc1c2nc(c3ccc(OC)cc3c2c4ccc(Cl)cc4)O

8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 ppm

wy-1-173c

13c

2014

11

28

166.69
161.40
160.60

147.44

132.79
131.65
131.26
130.86
129.96
129.04
128.85
126.18
118.84
118.67
118.07
110.80

77.85
77.85
76.75

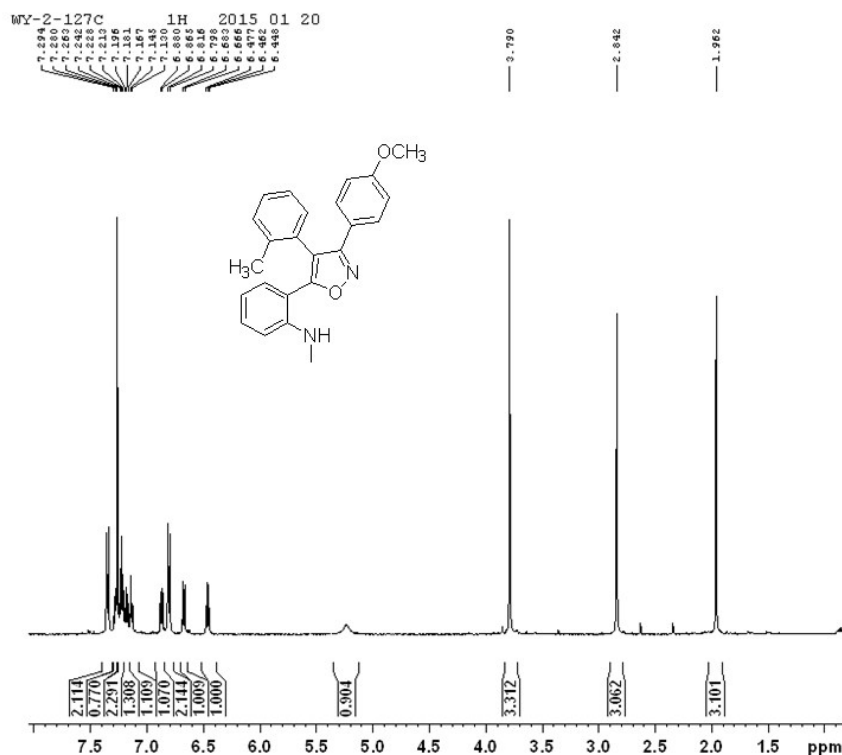
55.26

30.27

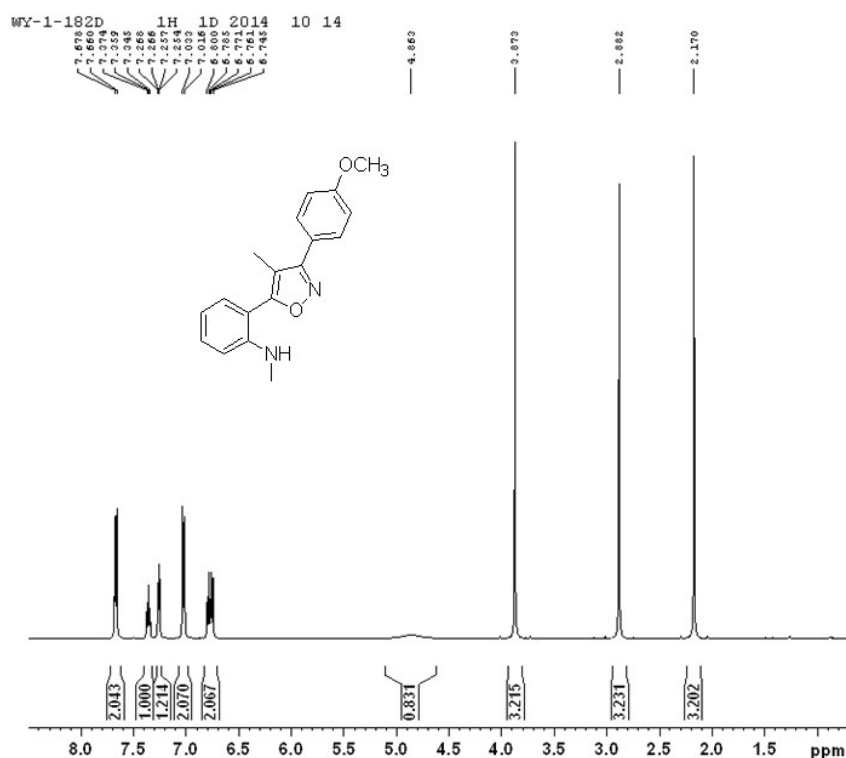
ppm

CNc1ccccc1C2=C(C(=O)N2C3=CC=C(C=C3)OC)C4=CC=C(C=C4)Cl

¹H NMR Spectrum of 4r



¹H NMR Spectrum of 4s

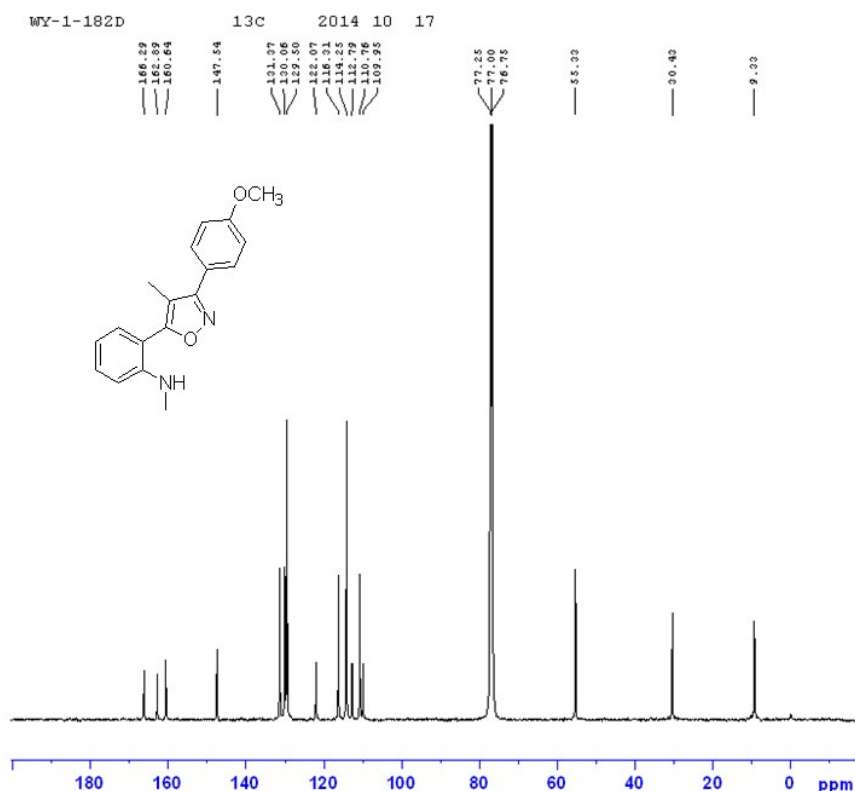


```

NAME      WY-1-182D
EXPNO     11
PROCNO    1
Date_     20141014
Time      14.21
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zg30
TD         16384
SOLVENT   CDCl3
NS         16
DS         1
SWH        10000.000 Hz
FIDRES     0.610352 Hz
AQ         0.8193000 sec
RG         912
DW         50.000 usec
DE         8.00 usec
TE         673.2 K
D1         1.0000000 sec
TD0        1

===== CHANNEL f1 =====
NUC1       1H
P1         13.00 usec
PL1        2.00 dB
SF01       500.0338500 MHz
SI         16384
SF         500.0300120 MHz
WDW        EM
SSB        0
LB         0.50 Hz
GB         0
PC         5.00
  
```

¹³C NMR Spectrum of 4s



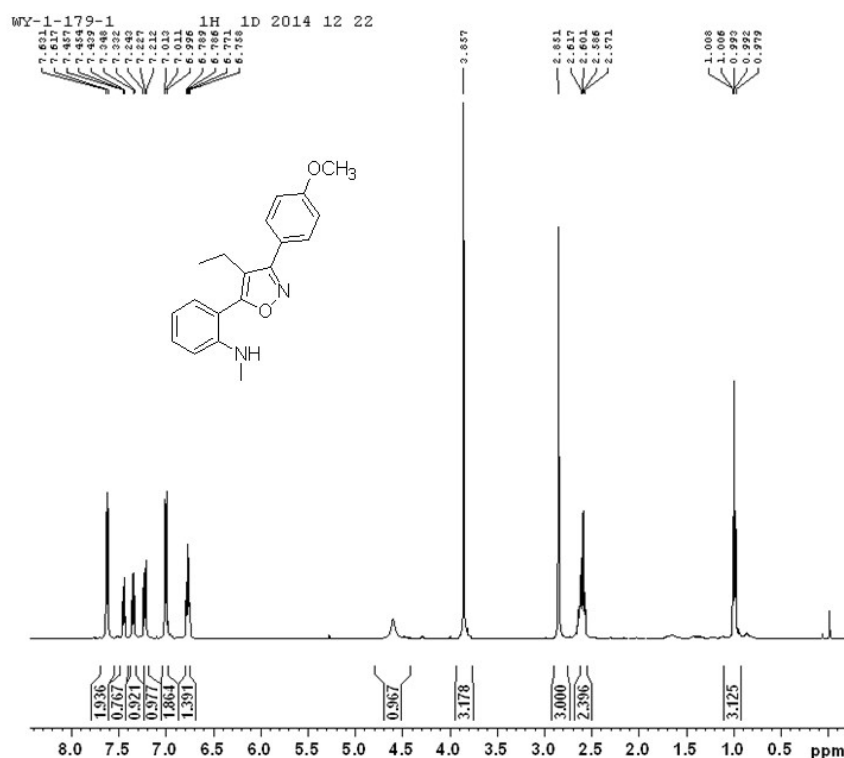
```

NAME      WY-1-182D
EXPNO     2
PROCNO    1
Date_     20141017
Time      22.14
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zgpg30
TD         65536
SOLVENT   CDCl3
NS         13607
DS         2
SWH        32679.738 Hz
FIDRES     0.498653 Hz
AQ         1.0027661 sec
RG         3250
DW         15.300 usec
DE         6.00 usec
TE         673.2 K
D1         2.0000000 sec
d11        0.0300000 sec
DELTA      1.89999998 sec
TD0        40

===== CHANNEL f1 =====
NUC1       13C
P1         12.20 usec
PL1        3.00 dB
SF01       125.7464750 MHz

===== CHANNEL f2 =====
CPDPRG2    waltz16
NUC2       1H
PCPD2      80.00 usec
PL2        2.00 dB
PL12       17.70 dB
PL13       17.70 dB
SF02       500.0355000 MHz
SI         32768
SF         125.7326441 MHz
WDW        EM
SSB        0
LB         12.00 Hz
GB         0
PC         2.00
  
```

¹H NMR Spectrum of 4t

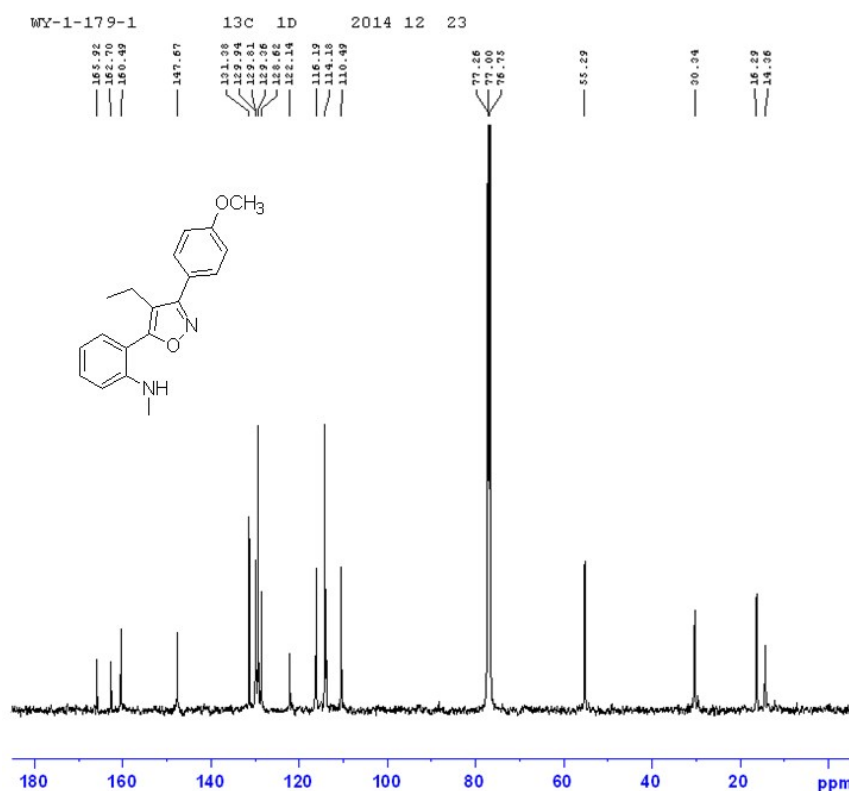


```

NAME      WY-1-179
EXPNO     1
PROCNO    1
Date_     20141222
Time      15.38
INSTRUM    spect
PROBHD     5 mm PABBO BB-
PULPROG    zg30
TD         16384
SOLVENT    CDCl3
NS         8
DS         1
SWH        10000.000 Hz
FIDRES     0.610352 Hz
AQ         0.8193000 sec
RG         144
DW         50.000 usec
DE         8.00 usec
TE         673.2 K
D1         1.00000000 sec
TD0        1

===== CHANNEL f1 =====
NUC1       1H
P1         13.00 usec
PL1        2.00 dB
SF01       500.0338500 MHz
SI         16384
SF         500.0300192 MHz
WDW        EM
SSB        0
LB         0.30 Hz
GB         0
PC         2.00
  
```

¹³C NMR Spectrum of 4t



```

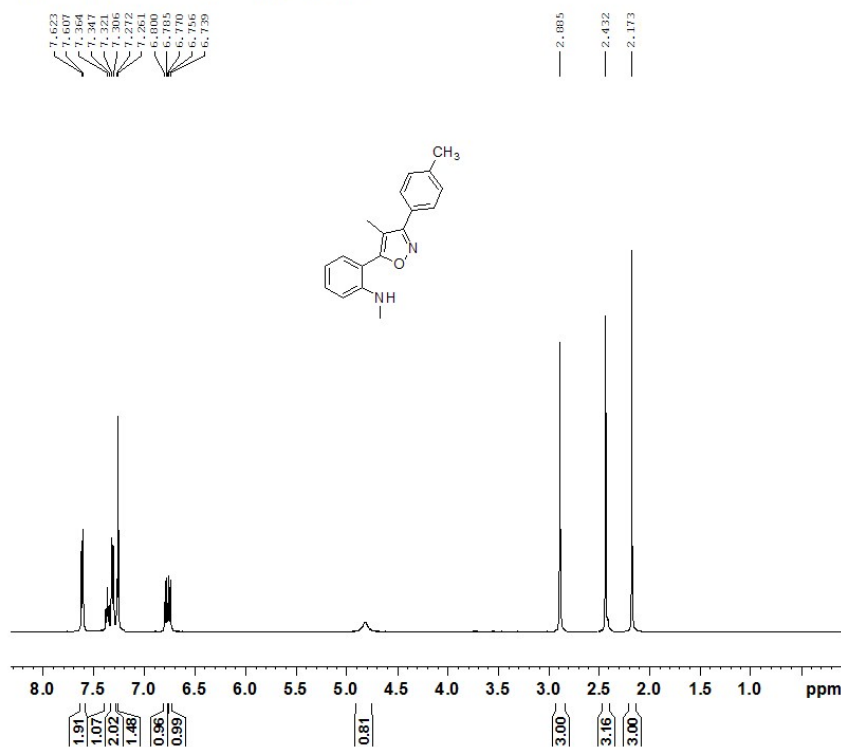
NAME      WY-1-179
EXPNO     2
PROCNO    1
Date_     20141223
Time      15.44
INSTRUM    spect
PROBHD     5 mm PABBO BB-
PULPROG    zgpg30
TD         65536
SOLVENT    CDCl3
NS         705
DS         2
SWH        32679.738 Hz
FIDRES     0.498653 Hz
AQ         1.0027661 sec
RG         1820
DW         15.300 usec
DE         6.00 usec
TE         673.2 K
D1         2.00000000 sec
d11        0.03000000 sec
DELTA      1.89999998 sec
TD0        20

===== CHANNEL f1 =====
NUC1       13C
P1         12.20 usec
PL1        3.00 dB
SF01       125.7464750 MHz

===== CHANNEL f2 =====
CPDPRG2    waltz16
NUC2       1H
PCPD2      80.00 usec
PL2        2.00 dB
PL12       17.70 dB
PL13       17.70 dB
SF02       500.0355000 MHz
SI         32768
SF         125.7326512 MHz
WDW        EM
SSB        0
LB         10.00 Hz
GB         0
PC         1.00
  
```

¹H NMR Spectrum of 4u

CAO-7-294 1H 2016 01 21

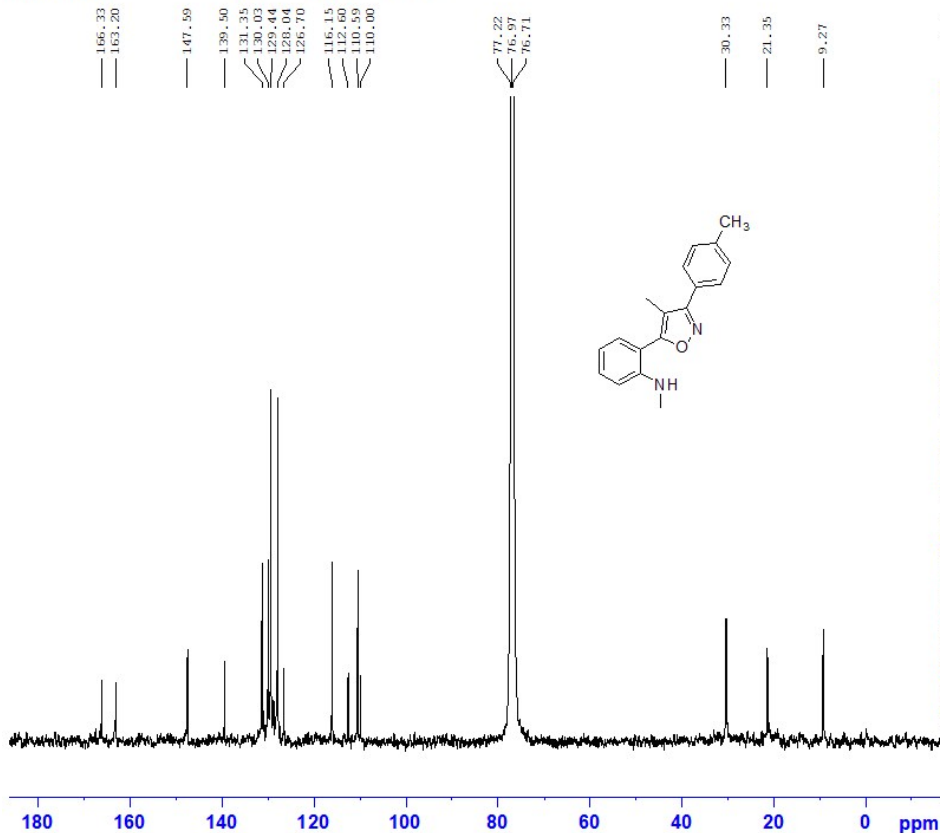


NAME CAO-7-294
EXPNO 1
PROCNO 1
Date 20160121
Time 15.29
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 16
DS 1
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 1.6385000 sec
RG 812
DW 50.000 usec
DE 6.00 usec
TE 673.2 K
D1 2.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 13.00 usec
PL1 2.00 dB
SFO1 500.0335010 MHz
SI 16384
SF 500.0300097 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
FC 4.00

¹³C NMR Spectrum of 4u

CAO-7-294 13C 2016 01 21



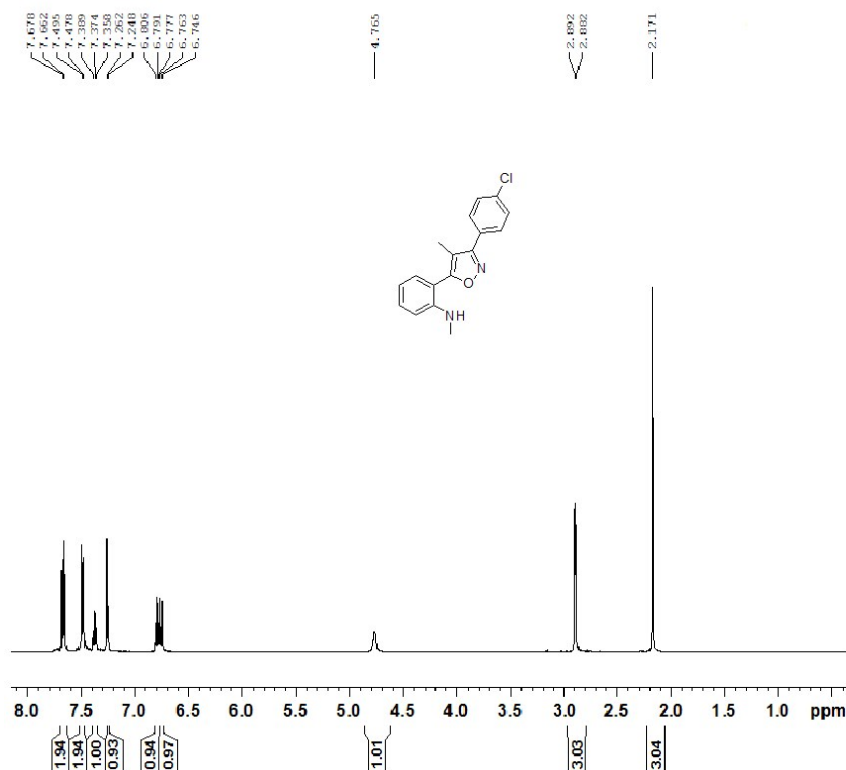
NAME CAO-7-294
EXPNO 1
PROCNO 1
Date 20160121
Time 22.57
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 13506
DS 2
SWH 32679.735 Hz
FIDRES 0.498653 Hz
AQ 1.0027661 sec
RG 3640
DW 15.300 usec
DE 6.00 usec
TE 673.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 20

===== CHANNEL f1 =====
NUC1 13C
P1 12.20 usec
PL1 3.00 dB
SFO1 125.7464750 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
FCFD2 80.00 usec
PL2 2.00 dB
PL12 17.70 dB
PL13 17.70 dB
SFO2 500.0355000 MHz
SI 32768
SF 125.7326487 MHz
WDW EM
SSB 0
LB 10.00 Hz
GB 0
FC 1.00

¹H NMR Spectrum of 4v

CAO-7-293 1H 2016 01 22

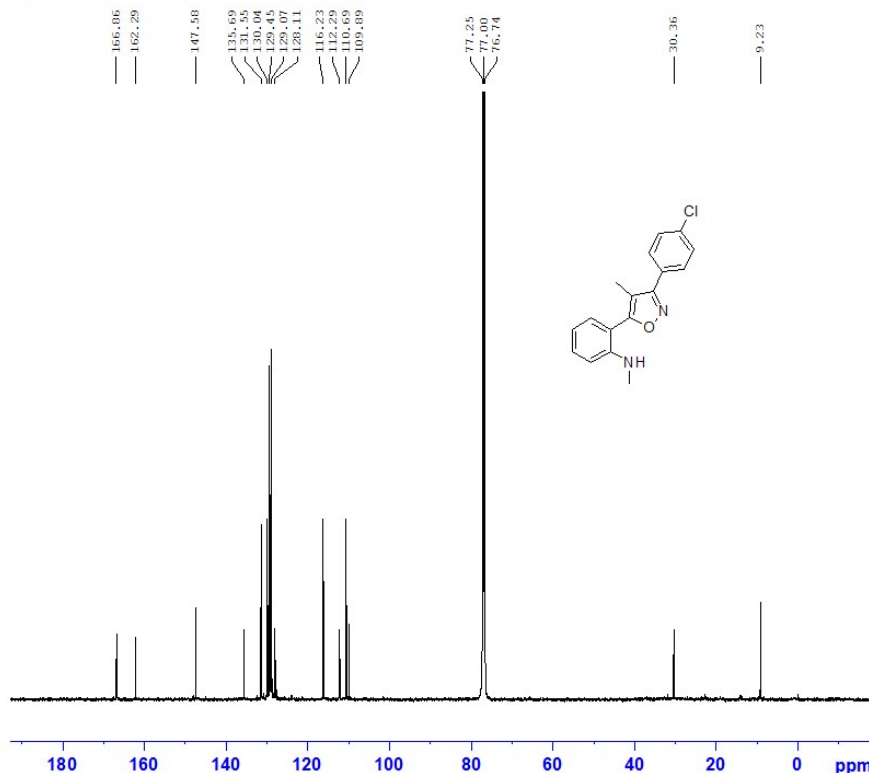


NAME CAO-7-293
EXPNO 1
PROCNO 1
Date_ 20160122
Time 17.34
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 8
DS 1
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 1.6385000 sec
RG 812
DW 50.000 usec
DE 6.00 usec
TE 673.2 K
D1 2.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 13.00 usec
PL1 2.00 dB
SFO1 500.035010 MHz
SI 16384
SF 500.0300097 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 4.00

¹³C NMR Spectrum of 4v

CAO-7-293 13C 2016 01 23



Current Data Parameters
NAME CAO-7-293
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20160123
Time 21.11
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 20000
DS 2
SWH 32679.738 Hz
FIDRES 0.498653 Hz
AQ 1.0027661 sec
RG 2300
DW 15.300 usec
DE 6.00 usec
TE 673.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 20

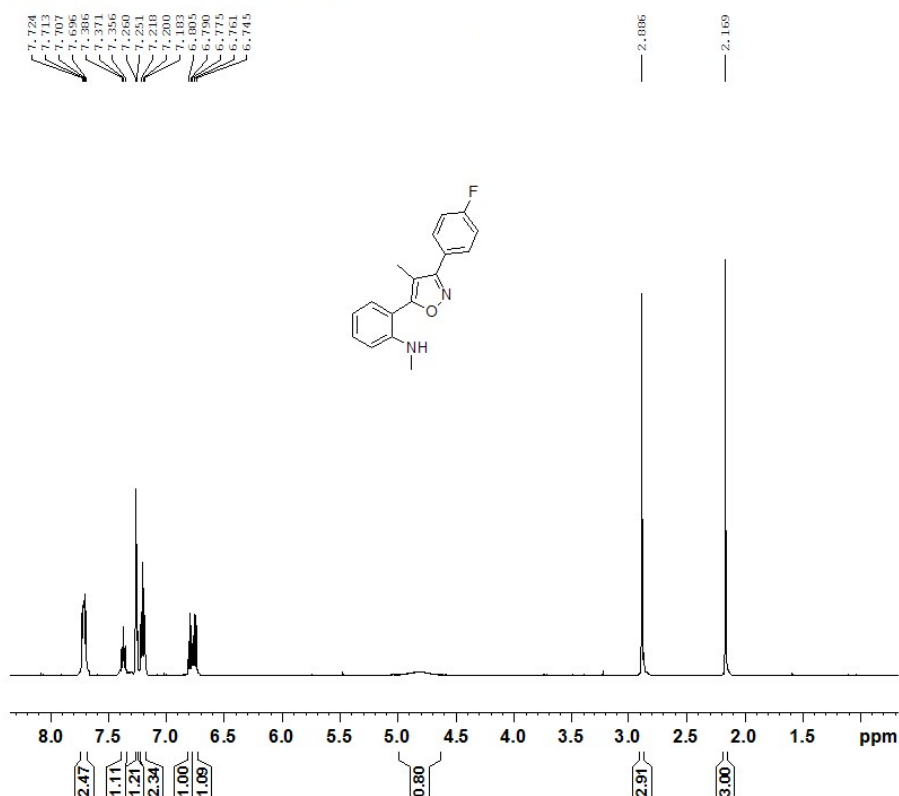
===== CHANNEL f1 =====
NUC1 13C
P1 12.20 usec
PL1 3.00 dB
SFO1 125.7464750 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.00 dB
PL12 17.70 dB
PL13 17.70 dB
SFO2 500.0355000 MHz

F2 - Processing parameters
SI 32768
SF 125.7326469 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00

¹H NMR Spectrum of 4w

CAO-7-298 1H 2016 01 27



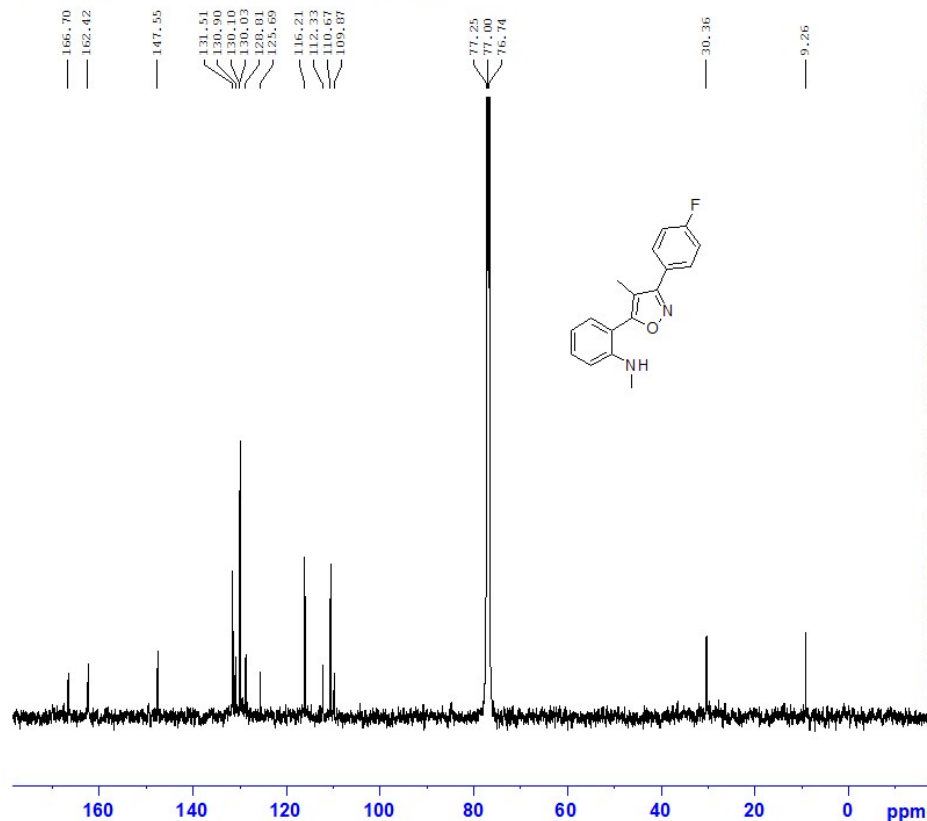
```

NAME          CAO-7-298
EXPNO         1
PROCNO        1
Date_         20160127
Time_         9.57
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       zg30
TD            32768
SOLVENT       CDCl3
NS            8
DS            1
SWH           10000.000 Hz
FIDRES        0.305176 Hz
AQ            1.6385000 sec
RG            406
DW            50.000 usec
DE            6.00 usec
TE            673.2 K
D1            1.00000000 sec
TD0           1

===== CHANNEL f1 =====
NUC1          1H
P1            13.00 usec
PL1           2.00 dB
SFO1          500.0335010 MHz
SI            16384
SF            500.0300102 MHz
WDW           EM
SSB           0
LB            0.30 Hz
GB            0
PC            4.00
  
```

¹³C NMR Spectrum of 4w

CAO-7-298 13C 2016 01 27



```

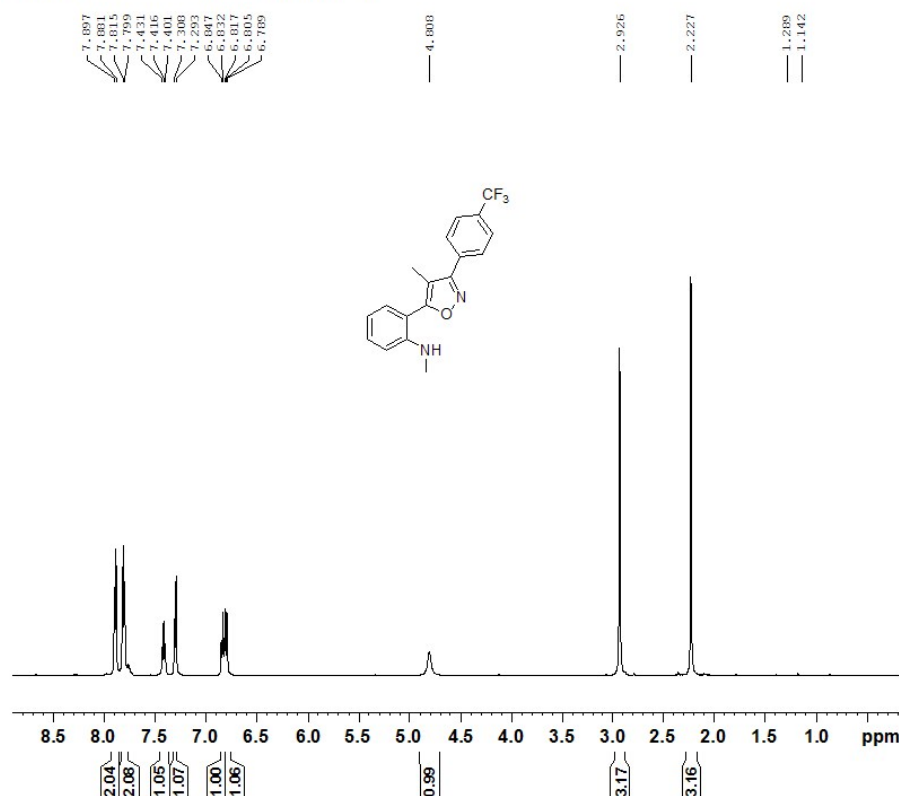
NAME          CAO-7-298
EXPNO         2
PROCNO        1
Date_         20160127
Time_         13.28
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       zgpg30
TD            65536
SOLVENT       CDCl3
NS            2281
DS            2
SWH           32679.738 Hz
FIDRES        0.498653 Hz
AQ            1.0027661 sec
RG            3640
DW            15.300 usec
DE            6.00 usec
TE            673.2 K
D1            2.00000000 sec
d11           0.03000000 sec
DELTA         1.89999998 sec
TD0           20

===== CHANNEL f1 =====
NUC1          13C
P1            12.20 usec
PL1           3.00 dB
SFO1          125.7464750 MHz

===== CHANNEL f2 =====
CPDPRG2       waltz16
NUC2          1H
PCPD2         80.00 usec
PL2           2.00 dB
PL12          17.70 dB
PL13          17.70 dB
SFO2          500.0355000 MHz
SI            32768
SF            125.7326487 MHz
WDW           EM
SSB           0
LB            6.00 Hz
GB            0
PC            1.00
  
```


¹H NMR Spectrum of 4x

CAO-7-300 1H 2016 02 04



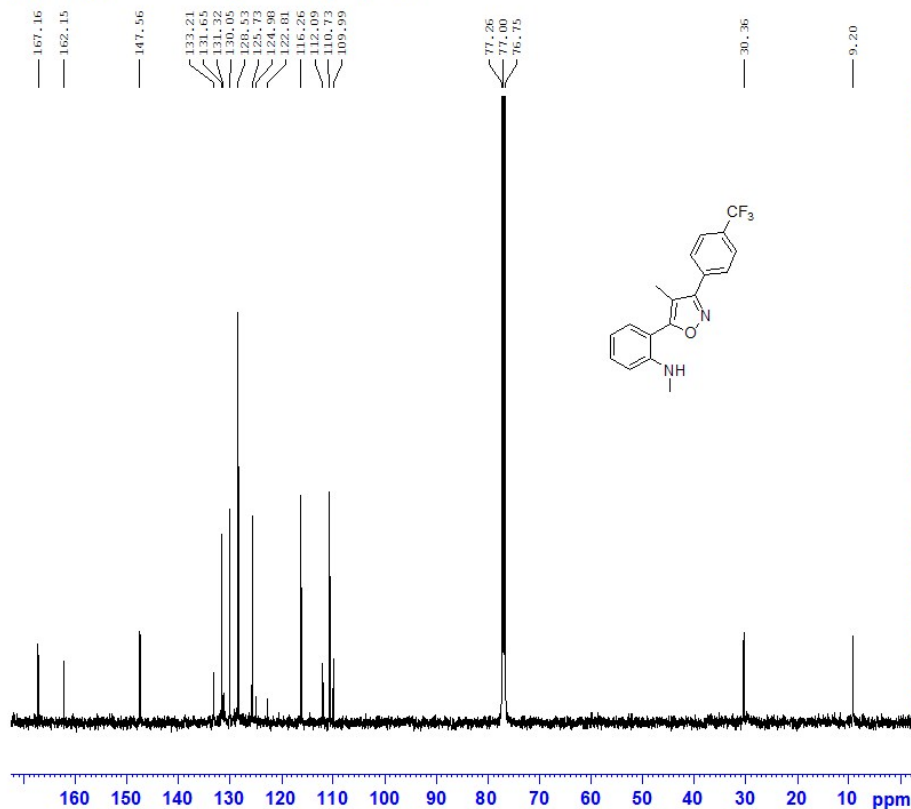
```

NAME      CAO-7-300
EXPNO     1
PROCNO    1
Date_     20160204
Time      12.31
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zg30
TD         32768
SOLVENT   CDCl3
NS         8
DS         1
SWH        10000.000 Hz
FIDRES     0.305176 Hz
AQ         1.6385000 sec
RG         228
DW         50.000 usec
DE         6.00 usec
TE         673.2 K
D1         1.00000000 sec
TD0        1

===== CHANNEL f1 =====
NUC1       1H
P1         13.00 usec
PL1        2.00 dB
SFO1       500.0335010 MHz
SI         16384
SF         500.0299937 MHz
WDW        EM
SSB        0
LB         0.30 Hz
GB         0
PC         4.00
    
```

¹³C NMR Spectrum of 4x

CAO-7-300 13C 2016 02 04



```

NAME      CAO-7-300
EXPNO     2
PROCNO    1
Date_     20160204
Time      12.47
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zgpg30
TD         65536
SOLVENT   CDCl3
NS         500
DS         2
SWH        32679.738 Hz
FIDRES     0.498633 Hz
AQ         1.0027661 sec
RG         3640
DW         15.300 usec
DE         6.00 usec
TE         673.2 K
D1         2.00000000 sec
d11        0.03000000 sec
DELTA     1.89999998 sec
TD0        20

===== CHANNEL f1 =====
NUC1       13C
P1         12.20 usec
PL1        3.00 dB
SFO1       125.7464750 MHz

===== CHANNEL f2 =====
CPDPRG2   waltz16
NUC2       1H
PCPD2      80.00 usec
PL2        2.00 dB
PL12       17.70 dB
PL13       17.70 dB
SFO2       500.0355000 MHz
SI         32768
SF         125.7326487 MHz
WDW        EM
SSB        0
LB         3.00 Hz
GB         0
PC         1.00
    
```